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FINAL

SAMPLING AND ANALYSIS PLAN

PHASE I REMEDIAL INVESTIGATION

R E M II

**PERFORMANCE OF REMEDIAL RESPONSE  
ACTIVITIES AT UNCONTROLLED  
HAZARDOUS WASTE SITES**

**U.S. EPA CONTRACT NO. 68-01-6939**

**CAMP DRESSER & MCKEE INC.**

**ROY F. WESTON, INC.**

**WOODWARD-CLYDE CONSULTANTS**

**CLEMENT ASSOCIATES, INC.**

**ICF INCORPORATED**

**C. C. JOHNSON & ASSOCIATES, INC.**

0000063

FINAL

SAMPLING AND ANALYSIS PLAN

PHASE I REMEDIAL INVESTIGATION

ORDOT LANDFILL, GUAM

Prepared by:

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DOCUMENT NO.: 279-RI1-OP-DXFL-1  
EPA WORK ASSIGNMENT NO: 168-9LA7.0  
EPA CONTRACT NO.: 68-01-6939

February 2, 1987

PERFORMANCE OF REMEDIAL RESPONSE ACTIVITIES  
AT UNCONTROLLED HAZARDOUS WASTE SITES (REM II)

U.S.EPA CONTRACT NO. 68-01-6939

SAMPLING AND ANALYSIS PLAN

FOR

PHASE I OF THE  
REMEDIAL INVESTIGATION

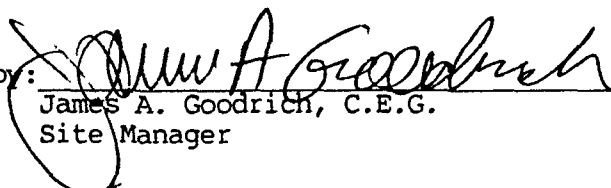
FOR

ORDOT LANDFILL, GUAM

EPA WORK ASSIGNMENT NO. 168-9LA7.0

REM II DOCUMENT CONTROL NO. 279-RI1-OP-DXFL-1

Approved by:

  
James A. Goodrich, C.E.G.  
Site Manager

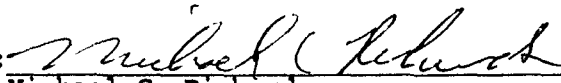
2-2-87  
Date

Approved by:

  
Sara R. Black, Peer Reviewer, Region IX-  
Technical Review Committee

2/3/87  
Date

Approved by:

  
Michael C. Richards  
Regional Manager

2/3/87  
Date

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U.S. ENVIRONMENTAL PROTECTION AGENCY REGION 9  
TOXICS & WASTE MANAGEMENT DIVISION  
FIELD OPERATIONS BRANCH

Sample Plan Title: Final SAP - Phase I Remedial Investigation  
Site Name: Ordot Landfill  
Site Location: Guam  
City/State/Zip: Ordot, Guam 96910  
Site EPA ID #: GUD - 980 - 637 - 649  
Anticipated Sampling Dates: March 16-20, 1987

Prepared by: Kevin E. Kelly/James A. Goodrich January 22, 1987  
Date  
Agency: Camp Dresser & McKee  
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EPA Project Officer: Thomas A. Mix (415) 974-8010  
Phone #

QAPP Approval Date: \_\_\_\_\_

\* *Transmit to \* FOR* \* \* \* \* \*  
*Expedited Review*  
F Received by Field Operations Branch: 2/3/87 \*  
O Date 2/3/87 \*  
R  
Received by Reviewer: 2/4/87 \*  
Date 2/4/87 \*  
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Date 2/4/87 \*  
E Concurrence: 2/4/87 \*  
P Chief, Field Operations Section Date 2/4/87 \*  
A Toxics & Waste Management Division  
Received by Quality Assurance Management Section: 2/4/87 \*  
Date 2/4/87 \*  
Analysis *Reviewed by:*  
U Reserved by: Patricia Connaughton \*  
S Laboratory Support Section Date 2/19/87 \*  
E Environmental Services Branch, OPM Date  
\* \* \* \* \*

## Summary of Analyses Requested

Site Name: ISLAND OF GUAM - ORDOT LANDFILLSample Plan Prepared by: JAMES A. GOODRICH/KEVIN E. KELLYEPA Project Officer: THOMAS A. MIXAnticipated Sampling Dates: MARCH 16-20, 1987

The totals for each category should come from the Tabular "Request for Analysis" in the sample plan.

## # OF ANALYSES

SAMPLE MEDIUM	ORGANIC ANALYSES							INORGANIC ANALYSES				SAMPLE TOTAL
	V	SV	P	ALL	D	SAS	TOTAL	M	CN	dist	SAS	
Low Conc. Soil	—	—	—	—	—	—	—	—	—	—	—	—
Medium Conc. Soil	—	—	—	—	—	—	—	—	—	—	—	—
Hi Conc. Soil	—	—	—	—	—	—	—	—	—	—	—	—
Low Conc. H <sub>2</sub> O	—	—	—	19	—	—	19	19	19	—	—	19
Medium Conc. H <sub>2</sub> O	—	—	—	—	—	—	—	—	—	—	—	—
Hi Conc. Liquid	—	—	—	—	—	—	—	—	—	—	—	—
OTHER	—	—	—	—	—	—	—	—	—	—	—	—

Specify other: \_\_\_\_\_

Specify SAS: 1. Analyses not CLP  
 2. Detection limit < CLP  
 3. Reduced turn around time

4. Dioxin/Furan (not 2,3,7,8-TCDD in soils)  
 5. Other \_\_\_\_\_

PROGRAM AUTHORIZATION: (CERCLA) RCRA FIFRA TSCA CWA

## PART I - SITE HISTORY AND BACKGROUND

The Island of Guam is located in the western Pacific region, approximately half-way between Japan and New Guinea, and is the largest island in the Mariana Island Group. Guam has an area of about 212 square miles, is approximately 30 miles long, and ranges between 4 and 11.5 miles wide (Figure 1). The island has two very distinct physiographic divisions. The southern half is composed of rugged volcanic upland and the northern half of the island is characterized by a limestone plateau. The majority of Guam's drinking water supply comes from groundwater produced from the sole-source limestone aquifer in the northern part of the island.

The Ordot Landfill is located in the northern part of the volcanic upland area, near the divide between the limestone and volcanic provinces (Figure 2). This divide consists of a fault and, as such, the exact demarcation between the two geologic units is not known. Furthermore, there may be some splays in the fault system which further complicate the exact delineation between the two units.

The landfill receives the majority of the wastes generated on the island and was designed and operated as a municipal landfill. However, the landfill is known to have received hazardous wastes during its history, which dates back to the Japanese occupation during World War II. The site is known to have received PCB contaminated oils from transformers, munitions, and hazardous wastes commonly used in households and light industry. However, records do not exist regarding when, how much, and what type of hazardous wastes were disposed of at the landfill.

Uncontrolled disposal of hazardous and other wastes at the Ordot Landfill has resulted in several problems, including uncontrolled surface water run-on and run-off from the site. The uncontrolled surface water run-on, including a large spring (Figure 2), which runs into and across the landfill, has resulted in leachate emanating from various locations around

the landfill. This leachate leaves the landfill site in the form of small springs or streams and eventually enters a stagnant pond or the Lonfit River (Figures 2 and 3). The leachate that discharges to the river eventually enters Pago Bay on the east side of the island, where fish kills have been reported. Contamination of marine life and recreational areas in Pago Bay are potential public health problems, therefore, it is proposed that leachate and surface water sampling be conducted near the site.

An additional possible public health problem due to the Ordot Landfill is the potential contamination of the sole-source aquifer in the area. As previously described, the Ordot Landfill is thought to be located in the southern physiographic province where volcanic bedrock exists. The exact demarcation between the volcanics and the limestone of the north is not known. Due to the proximity of the landfill to the limestone unit, there is some concern that there is a potential for contaminating the limestone aquifer. There is also some concern that a portion of the landfill may directly overlay the limestone aquifer, in which case there would be a definite public health problem. Due to these circumstances, limited geologic reconnaissance and groundwater sampling efforts are proposed for the area.

In addition to the hydrologic concerns, there has historically been an air quality problem due to noxious odors, caused in part, by the presence of dead animals. In order to accurately characterize this problem, a limited, reconnaissance-level air quality data collection program is proposed. This program includes the collection of data with field instruments. The program is discussed in greater detail in latter sections.

PART II - MAPS

- Figure 1      Location of Ordot Landfill - Island of Guam
- Figure 2      Ordot Landfill Site Map
- Figure 3      Location of Surface Water, Leachate, and Reconnaissance  
Geologic Sampling Locations

## PART III - OBJECTIVE OF SAMPLING EFFORT

### 3.1 STATED OBJECTIVE OF SAMPLING EFFORT

The primary objective of the Phase I Investigation (Initial Site Characterization), hereafter referred to as the "ISC," is to determine if contaminants are present at the site and migrating off site; identify specific contaminants as well as identify contaminant concentrations, quantities, and physical states. In order to meet these objectives, the following activities will be performed at the site:

1. Determine the quality of leachate leaving the boundaries of the landfill;
2. Determine the water quality in the Lonfit River, both upstream and downstream of the landfill;
3. Determine the water quality of the groundwater in the limestone aquifer in the vicinity of the town of Ordot;
4. Perform a reconnaissance-level geologic investigation in the vicinity of the landfill in order to identify the bedrock unit underlying the landfill; and
5. Perform a reconnaissance-level air quality survey to portray field conditions.

The data collected from these field efforts will be utilized to determine whether additional site characterization is necessary. For example, the data will be utilized to determine the level of contaminants at the boundary of the site, establish potential pathways, establish the nature of the contaminants that are leaving the source, and define the potential for exposure to surrounding populations. Following these evaluations, a determination will be made with respect to the focus of additional efforts and the objectives of the Phase II Investigation. In regard to the air quality data collection effort, these data will assist in establishing levels of protection required for site personnel and if additional data collection efforts should be considered as a part of Phase II studies.

PART IV - RATIONALE FOR SAMPLE LOCATIONS,  
NUMBER OF SAMPLES, AND ANALYTICAL PARAMETERS

4.1 INTRODUCTION

As indicated above, several potential health problems exist due to poor surface runoff control present at the Ordot Landfill. These poor controls could potentially result in the contamination of both groundwater and surface water supplies in the area. In order to better understand the potential for contamination, an ISC has been proposed for the area. This ISC will result in the collection of (1) leachate samples, (2) surface water samples, (3) a groundwater sample, and (4) qualitative air data. In addition, a reconnaissance level geologic investigation will be conducted in order to define the bedrock surface in the area. As previously discussed, the exact location of the fault separating the two geologic units is not well defined. As such, the demarcation of the fault is important in defining the geology underlying the landfill and the potential for contaminating the limestone aquifer. This information will be utilized to develop a preliminary assessment for the site.

The specific rationale for each sample site is provided in Table 4-1. As discussed in Part V, it is anticipated that each water sample will be analyzed by the Contract Laboratory Program (CLP) for the complete Target Compound List (TCL) organic and inorganic analyses included in the Routine Analytical Service (RAS). Air quality data will be collected using field instruments and, as such, analytical support will not be requested. The rationale for selecting the complete RAS analyses is that there is not an extensive water quality data base for the area. Furthermore, the development of a data base which characterizes the leachate, surface water, groundwater, and air quality will be important for additional field efforts to be conducted under the Phase II Investigation. The rationale for the collection of each media is presented below.



TABLE 4-1

## RATIONALE FOR SAMPLING LOCATION

Sample Location	Sample Number	Rationale
SW-1 <sup>a</sup>	279-SW-001-001	Determine water quality in Lonfit River upstream of Ordot Landfill
SW-2 <sup>a</sup>	279-SW-002-001	Determine water quality in Lonfit River downstream of Ordot Landfill
SW-3 <sup>a</sup>	279-SW-003-001	Determine quality of leachate discharge emanating from west side of landfill
SW-4 <sup>a</sup>	279-SW-004-001	Determine quality of leachate discharge from southwest side of landfill
SW-5 through SW-10 <sup>a</sup>	279-SW-005-001 279-SW-006-001 279-SW-007-001 279-SW-008-001 279-SW-009-001 279-SW-010-001	Determine quality of leachate discharges from various locations along south side of landfill
SW-11 <sup>a</sup>	279-SW-011-001	Determine water quality of spring previous to discharge into landfill
SW-12 <sup>a</sup>	279-SW-012-001	Determine water quality of pond where leachate collects
GW-01 <sup>b</sup>	279-GW-001-001	Determine groundwater quality in vicinity of landfill

<sup>a</sup> Refer to Figure 3 for sampling locations

<sup>b</sup> Refer to Figure 2 for sampling location

#### 4.2 LEACHATE SAMPLE COLLECTION

A fault separates the limestone and volcanic provinces in Guam (as shown on Figure 2). A spring issues near the fault along the northeastern boundary of the site and flows southwest into and beneath the landfill and emerges on the south side of the site, where it flows into the Lonfit River. Flow from this spring varies according to the season. During a recent site visit, which was in the wet season, flow was at least 100 gallons per minute (gpm). The spring water flowing from beneath the landfill was foaming during the site visit. Leachate, probably derived from rainfall infiltrating through the landfill, flows out of the landfill around its perimeter. As a result, the valley bottom immediately south of the site is in a wetlands condition. Some leachate streams contain gas bubbles and grease, as well as foam.

As discussed in Section 3.2, there is a possibility that some hazardous materials have been deposited in the landfill. For this reason, it is proposed that these leachate streams be sampled. Ten (10) samples will be collected. Approximately eight (8) leachate streams identified during the recent site visit are included in this sampling plan. In addition, one (1) sample will be collected from the spring prior to its flow into the landfill. One (1) sample will also be collected from the pond where the leachate apparently collects. These data will be utilized to develop the potential effect this landfill may have on the Lonfit River, as well as Pago Bay.

#### 4.3 SURFACE WATER SAMPLE COLLECTION

It is proposed that two (2) surface water samples will be collected along the Lonfit River. One sample will be collected upstream of the landfill and one sample will be collected directly downstream of the landfill. The locations are shown on Figure 3. The rationale for collecting these samples is to provide background water quality data for the Lonfit River at the upstream sampling station, as well as collecting data to evaluate the potential effect of the landfill on the downstream station. This type of

data will assist in refining the scope of work for the RI with respect to off-site releases from the landfill and in assessing potential health effects. Although this is only one sampling effort, it will provide data which will assist in defining the scope required for the RI.

#### 4.4 GROUNDWATER SAMPLE COLLECTION/GEOLOGIC RECONNAISSANCE

Although the Ordot Landfill site is apparently located in the volcanic region of Guam, it is immediately adjacent to the limestone area to the north, which is the sole-source aquifer for the island. Air photo analysis and slope debris suggest that limestone may underlie the site, but this has not been substantiated. If limestone underlies the site, then leachate from the site may be moving north into the sole source limestone aquifer of northern Guam. To substantiate this potential, a groundwater sample will be collected from the Washington Jr. High School Well (Figure 2). This well is the only groundwater sampling point in the sampling vicinity. As such, this will provide valuable data and assist in determining the potential for the off-site excursion of contaminants. In conjunction with the groundwater sample collection, a reconnaissance-level investigation of the bedrock in the area of the landfill will be conducted. The investigation will involve excavating trenches with a bulldozer to expose the bedrock surface. The locations of these trenches are shown on Figure 3. The rationale for performing this investigation is to determine the composition of the bedrock surface and to evaluate the potential for the landfill to be hydraulically connected to the sole-source limestone aquifer located to the north.

#### 4.5 AIR QUALITY RECONNAISSANCE

Historically, noxious odors have been a problem at the Ordot landfill. In part, these odors have been caused by dead domestic animals discarded at the landfill. These odors are magnified by the decay of waste in the hot and humid tropical environment. In addition, historic subterranean fires indicate the production of flammabel gasses within the landfill. In order to provide some preliminary reconnaissance-level data on the air quality at the site, the sampling personnel will be utilizing field instruments.

These field instruments will include a Century Portable Organic Vapor Analyzer (OVA), a HNu Photoionization Detector, a Ludlum Radiation Meter, and a Gastechtor Hydrocarbon Survey instrument. These instruments are also required as a part of the Health and Safety Plan to ensure the protection of field personnel during the leachate, surface water and groundwater sample collection efforts.

The approach to the study will be to traverse the site, as well as the boundaries of the site, with the instruments to identify the presence of emissions from the landfill. The proposed suite of instruments will allow for the detection of organic emissions (OVA and HNu) and methane (Gastechtor). Specific areas to be traversed will be identified through site inspection, as well as interviews with site workers and local health and regulatory officials. Although this approach will not allow quantification of contaminants, these preliminary data will indicate whether emissions are a potential problem. If significant emissions are detected and identified, then a more focused and detailed air quality monitoring program can be developed and implemented during Phase II studies.

TABLE 4-2

Typical Military Landfill Installations And  
Common Organic Compounds Disposed There

<u>Installation</u>	<u>Compounds</u>
Touele, Utah	1,1 Dichloroethane; Tetrachloroethylene; Trichloroethylene; Methylene Chloride; Chloroform
Hill Air Force Base, Utah	Methyl Ethyl Ketone, Trichloroethane, Carbon Tetrachloride, Benzene, Dichlorobenzene, Chlorobenzene, Toluene, Xylene, Phenol, Ethyl Benzene
Rocky Mountain Arsenal, Colorado	Bromoform; 1,2 Dichloroethylene; 1,1 Dichloroethylene Benzanthracene, Benzo(a)Pyrene, Chrysene
Ordot, Guam	PCB containing Transformer Oils; Munitions; Household and Light Industry Hazardous Waste; Acetone; Methanol; Pesticides; Herbicides; Naphthalene

SOURCE 208 PLAN FOR  
THE ISLAND OF GUAM

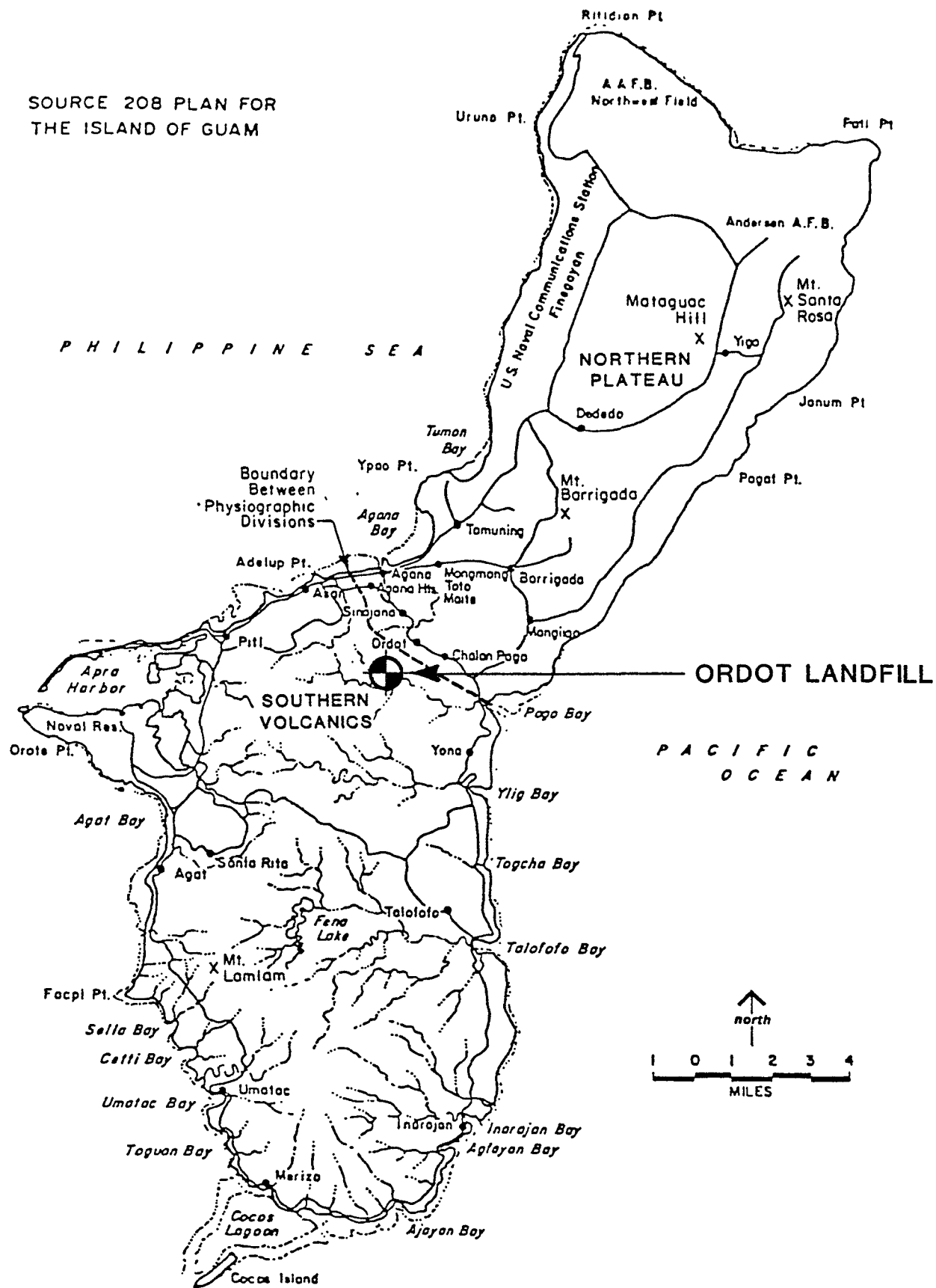
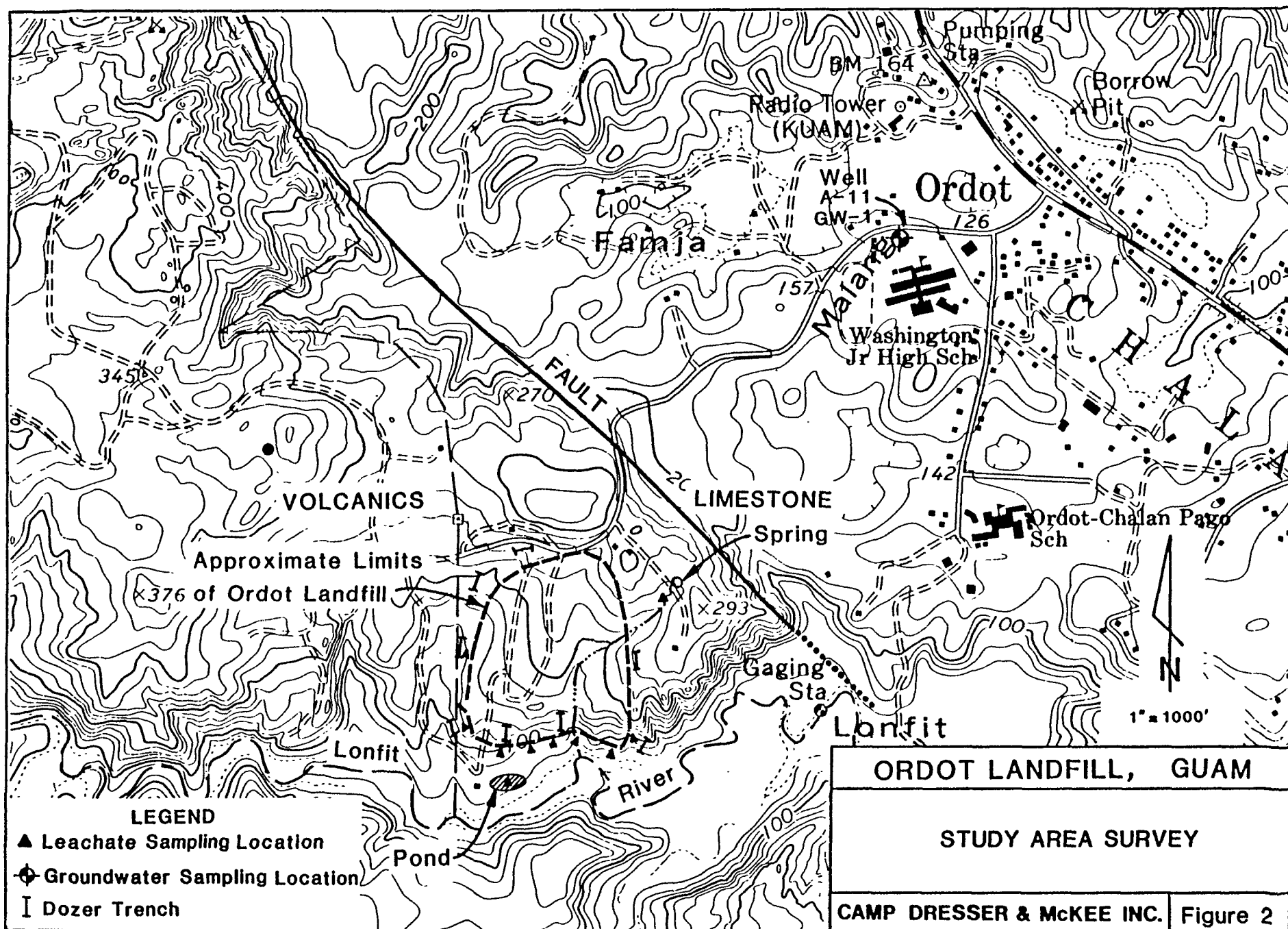
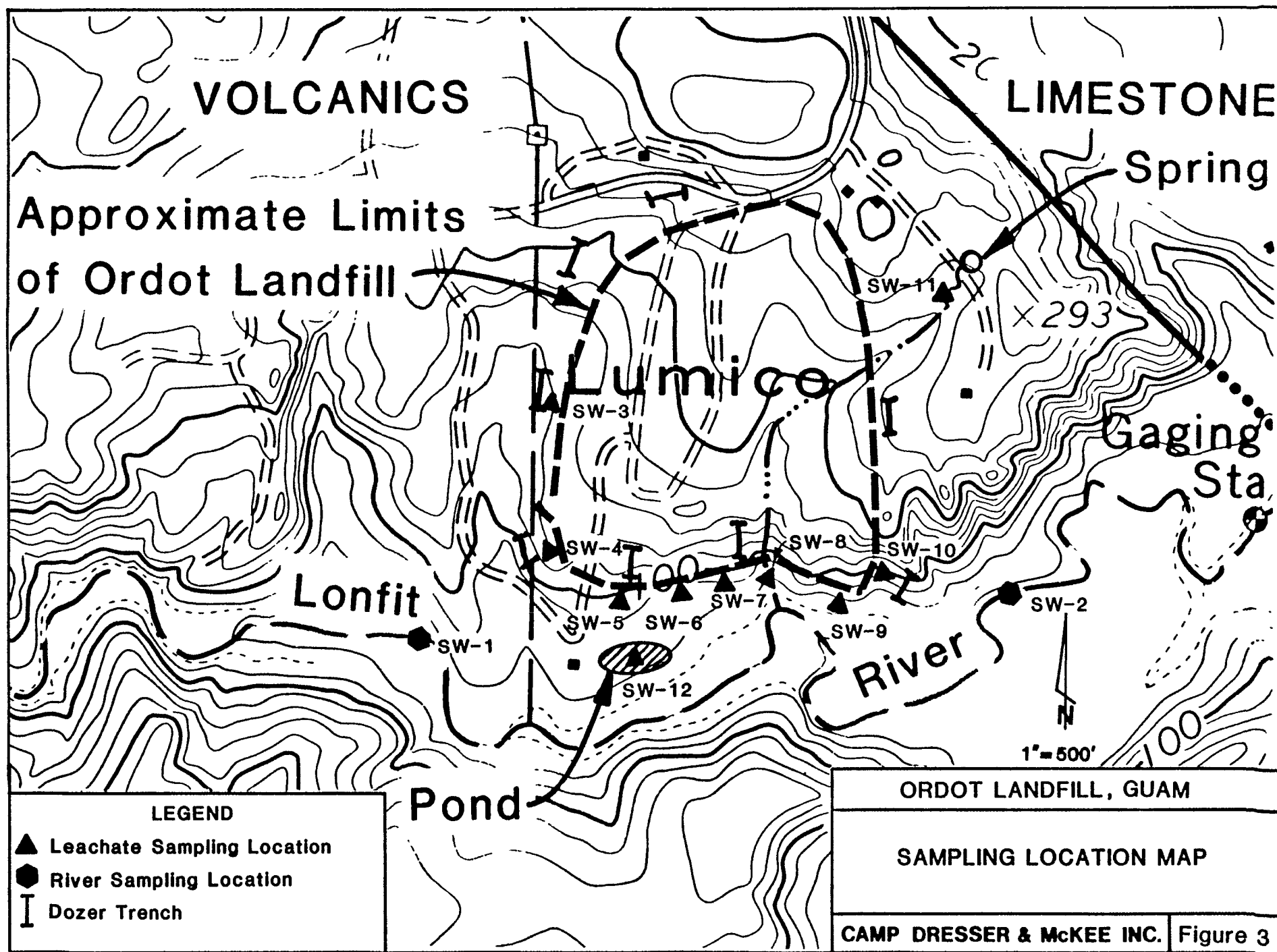


FIGURE 1  
LOCATION OF ORDOT LANDFILL  
ISLAND OF GUAM







## REQUEST FOR WATER SAMPLE ANALYSIS (by sample)

Location	Proposed Date	Concentration			Analysis Required						
		Low	Med	High	Organics <sup>b</sup>				Inorganics <sup>c</sup>		Other <sup>d</sup>
GW -1	Day 1	X			V	SV	P	All	M	CN-dist	
Duplicate (GW-1)	Day 1	X			V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
Totals:	Low Concentration							2	2	2	
	Medium Concentration:										
	High Concentration:										

The above totals should also be recorded on the "Summary of Analyses Requested"

a.

	<u>LOW</u>	<u>MEDIUM</u>	<u>HIGH</u>
<u>Concentration:</u>	< 10 ppm	10 ppm to 15 % of any 1 constituent	15 % to 100 % of any 1 constituent

b. Organics Analysis: V = volatiles, SV = semivolatiles, P = pesticides/PCBs;  
All = V + SV + P.

c. Inorganics Analysis: M = metals, CN-dist = distillation method for cyanides.

d. Identify Special Analytical Services (SAS) requested.

## REQUEST FOR WATER SAMPLE ANALYSIS (by sample)

[illegible]

The above totals should also be recorded on the "Summary of Analyses Requested"

**2.**

	<u>LOW</u>	<u>MEDIUM</u>	<u>HIGH</u>
<u>Concentration:</u>	< 10 ppm	10 ppm to 15 % of any 1 constituent	15 % to 100 % of any 1 constituent

b. Organics Analysis: V = volatiles, SV = semivolatiles, P = pesticides/PCBs;  
All = V + SV + P.

c. Inorganics Analysis: M = metals, CN-dist = distillation method for cyanides.

**d. Identify Special Analytical Services (SAS) requested.**

## REQUEST FOR WATER SAMPLE ANALYSIS (by sample)

Location	Proposed Date	Concentration			Organics <sup>b</sup>				Inorganics <sup>c</sup>		Other <sup>d</sup>
		Low	Med	High	V	SV	P	All	M	CN-dist	
SW-3	Day 2	X			V	SV	P	All	M	CN-dist	
SW-4	Day 2	X			V	SV	P	All	M	CN-dist	
SW-5	Day 2	X			V	SV	P	All	M	CN-dist	
SW-6	Day 2	X			V	SV	P	All	M	CN-dist	
SW-7	Day 2	X			V	SV	P	All	M	CN-dist	
Duplicate (SW-6)	Day 2	X			V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
					V	SV	P	All	M	CN-dist	
Totals:		Low Concentration						6	6	6	
		Medium Concentration:									
		High Concentration:									

The above totals should also be recorded on the "Summary of Analyses Requested"

a.

	LOW	MEDIUM	HIGH
Concentration:	< 10 ppm	10 ppm to 15 % of any 1 constituent	15 % to 100 % of any 1 constituent

b. Organics Analysis: V = volatiles, SV = semivolatiles, P = pesticides/PCBs;  
All = V + SV + P.

c. Inorganics Analysis: M = metals, CN-dist = distillation method for cyanides.

d. Identify Special Analytical Services (SAS) requested.

## REQUEST FOR WATER SAMPLE ANALYSIS (by sample)

Sample	Proposed Date	Concentration <sup>a</sup>			Analysis Requested							Other <sup>d</sup>
		Low	Med	High	Organics <sup>b</sup>				Inorganics <sup>c</sup>			
SW-8	Day3	X			V	SV	P	All	M	QN-dist		
SW-9	Day3	X			V	SV	P	All	M	QN-dist		
SW-10	Day3	X			V	SV	P	All	M	QN-dist		
SW-11	Day3	X			V	SV	P	All	M	QN-dist		
SW-12	Day3	X			V	SV	P	All	M	QN-dist		
Duplicate (SW-12)	Day3	X			V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
Travel Blank	Day3	X			V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
					V	SV	P	All	M	QN-dist		
Totals:	Low Concentration							7	7	7		
	Medium Concentrations:											
	High Concentrations:											

The above totals should also be recorded on the "Summary of Analyses Requested"

a.

	LOW	MEDIUM	HIGH
Concentration:	< 10 ppm	10 ppm to 15 % of any 1 constituent	15 % to 100 % of any 1 constituent

b. Organics Analysis: V = volatiles, SV = semivolatiles, P = pesticides/PCBs;  
All = V + SV + P.

c. Inorganics Analysis: M = metals, QN-dist = distillation method for cyanides.

d. Identify Special Analytical Services (SAS) requested.

## PART V - REQUEST FOR ANALYSES

This section presents request for analysis. It is anticipated that 19 RAS water samples will be collected during the ISC effort. These include the following:

- o Eight (8) leachate stream samples around the landfill (locations SW-3 through SW-10; Figure 3) .
- o Two (2) surface water samples from the Lonfit River (locations SW-1 and SW-2; Figure 3).
- o One (1) spring sample (location SW-11; Figure 3).
- o One (1) leachate pond sample (location SW-12; Figure 3).
- o One (1) groundwater sample from a well located at Washington Jr. High School (Figure 2).
- o Three duplicates including (1) one groundwater sample, (2) one surface water sample, and (3) one leachate sample.
- o Three travel blanks, one for each day of anticipated sample shipment.

Due to the anticipated similarity of the spring, pond, and surface water, one duplicate will be collected for these sample types.

Each sample will be analyzed for Target Compound List (TCL) volatile, semi-volatile, pesticide/PCB, and metal parameters under the RAS program.

Rinsate blanks for the water sampling effort will not be included since field equipment will not be utilized more than once for the sampling. For example, a large disposable sample container will be used to collect the

leachate surface water samples which will be aliquoted into the sample bottles. Only one groundwater sample will be collected from the well, and therefore, the bailer will only be used once.

These samples are summarized in the Sample Analysis forms. The sample collection sites are shown in Figures 2 and 3.

## PART VI - SAMPLING METHODS AND PROCEDURES

### 6.1 INTRODUCTION

As previously described, the ISC will involve the following activities: (1) surface water/leachate sample collection, (2) groundwater sample collection, (3) qualitative air data collection, and (4) geologic reconnaissance. The detailed procedures for performing these activities are described in CDM's Site Investigation Procedures Manual (SIPM). The appropriate procedures are reproduced and presented in the Appendix. A brief discussion of the procedures associated with each ISC activity is presented below. In addition, the procedures associated with sample handling, shipping, identification, etc., and the quality assurance aspects are discussed below.

### 6.2 GENERAL SAMPLING PROCEDURES

The general sampling procedures include sample classification, handling, and shipment; sample identification; and sample bottle preparation. The SIPM method numbers are presented in Table 6-1. Each of these methods is presented in the Appendix. In addition, Table 6-2 provides information regarding the containers, volumes, preservatives, and holding times for the samples.

#### 6.2.1 Sample Classification, Handling and Shipping

Classification - The protection of personnel involved in the shipment of samples to contract laboratories is important, as well as the maintenance of the integrity of the samples. When sent by common carrier, the packaging, labeling, and shipping of hazardous wastes and substances are regulated by the U.S. Department of Transportation (DOT) under 49 CFR Parts 171-173.

Samples obtained at uncontrolled hazardous waste sites are classified as either environmental samples or hazardous samples. Environmental samples are those which contain low levels of contaminants and require implementa-

TABLE 6-1  
GENERAL SAMPLING PROCEDURES

Procedure	Method Number
Procedure for Use and Maintenance of Field Notebooks	5621004
Sample Classification, Handling and Shipment	5622001
Sample Identification Procedure	5622002
Sample Bottle Preparation, Sample Preservation, and Maximum Hold Times	5622006

tion of limited precautionary procedures. Hazardous samples are those which could possibly contain dangerous levels of contaminants. Hazardous samples must be packaged and labeled according to procedures specified by the U.S. DOT, or the Guam DOT, whichever is more stringent. For the purpose of this ISC, the samples collected around the site will be considered environmental. The leachate samples will also be screened with a Radiation Meter for readings for alpha, beta, and gamma, according to SIPM procedure number 5625001.

Environmental samples are collected in an appropriate container allowing approximately 10 percent air space, with the exception of volatile organics, so that the container is not full at 130°F. The sealed and labeled container is then placed inside a ziplock polyethylene bag which is also sealed. The sealed package is then placed inside a shipping container, packed so as to prevent breakage. No precautionary notices are required on the package exterior, although "Environmental Laboratory Samples" and "This Side Up" labels will be placed on the shipping containers.

Packaging - As provided in Table 6-2, each water sample will be collected in the following containers: two (2) 40-milliliter volatile organic glass vials with teflon septums, four (4) 1-liter amber glass bottles with teflon



TABLE 6-2

CONTAINERS, VOLUMES, PRESERVATIVES, AND HOLDING TIMES FOR  
PARAMETERS INCLUDED IN ISC

RAS Parameter	Container	Volume Required	Preservative	Holding Time
TCL Volatile Compounds	Two (2) - 40 ml Glass Vials with Teflon-lined Septum Cap	80 ml	Add 2 drops 1:1 HCL per vial before sample collection, Cool to 4°C	Analyze within 10 days
TCL Semi-Volatile and Pesticide Compounds	Four (4) 1-Liter Amber Glass Bottles with teflon-lined poly caps	1 Gallon	Cool to 4°C	5 days until extraction, analyze within 40 days after extraction
Inorganic Dissolved Metals (Except Cyanide) for Ground Water	One (1) 1-Liter Polyethylene Bottle with poly cap	1 Liter	Field Filtered, HNO <sub>3</sub> to pH ≤ 2, Cool to 4°C	6 months
Inorganic Total Metals (Except Cyanide) Leachate/Surface Water	One (1) 1-Liter Polyethylene Bottle with poly cap	1 Liter	HNO <sub>3</sub> to pH ≤ 2, Cool to 4°C	6 months
Cyanide (Included with RAS Metals)	One (1) 1-Liter Polyethylene Bottle with poly cap	1 Liter	NaOH to pH > 12, 0.6 g ascorbic acid (in presence of residual chlorine), Cool to 4°C	14 days

lined screw caps, and two 1-liter polyethylene bottles. Each container will have a chain-of-custody seal placed over the cap and attached to the sample, although the VOA vials will be placed in a bag with seals placed on the bag. The samples will then be placed in a DOT-approved shipping container, such as a Coleman ice chest. Vermiculite will be placed around each container to prevent breakage during shipment. In addition, ice packs will be placed in the ice chest to comply with preservation requirements, which require the cooling of most of the samples to 4° Celsius. These ice packs will maintain the required temperature for a number of days.

The water sample containers for conducting this field effort will be obtained from the CLP Sample Bottle Repository. This request will be made through the official requestor. Arrangements will be made to ship the containers to Guam. All references made in SIPM 5622006 regarding obtaining, cleaning, etc. of water sample containers will not be applicable since these containers will be received from the CLP.

In addition, a complete chain-of-custody record, as well as other appropriate documentation, will be sealed in a plastic or ziploc bag and placed in the container. These procedures are described in Section 6.7.

Transportation - All samples will be shipped out of Guam by commercial aircraft, such as Continental Airlines, which will provide airport to door service. An additional service available is the DHL Worldwide Express, which provides door to door service. DHL guarantees 3 day service, through customs, from Guam to any place in the United States. Environmental samples also may be transported by REM II personnel in private vehicles. The samples will be shipped each day following the collection and documentation procedures.

#### 6.2.2 Sample Identification

Sample Identification - A coding system is used to identify each sample taken during the sampling program. This coding system will provide a tracking procedure to allow retrieval of information concerning a particular sample and assure that each sample is uniquely identified. The sample

identification procedures for the surface water and groundwater samples are provided in this section.

The sample identification number will consist of a three-letter designation to identify the site, which will be 279 in this case. A two-letter designation follows the site designation and will be used to identify the type of sampling media:

GW - Groundwater Samples

SW - Surface Water/Leachate Samples

The letter designations are followed by three digits, which indicate the station location. The leachate surface water station locations are provided in Figure 3. Figure 2 provides the groundwater sample collection location. The final number provides the sequential number for samples collected at that station location. Therefore, a typical sample number will be:

<u>Project Code</u>	<u>Sample Media</u>	<u>Station No.</u>	<u>Sequential Number</u>
279	GW	001	001

or 279-GW-001-001, indicating that this is the first groundwater sample collected from Site No. 1 from the Ordot Landfill site. Locations where air quality measurements are performed will be noted on a map of the site, with the field data entered into the field logbook.

### 6.3 SURFACE WATER/LEACHATE SAMPLE COLLECTION

As previously mentioned, two (2) surface water and ten (10) leachate samples will be collected from various locations around the landfill. The locations of these sites are provided on Figure 3. The sample designations and rationale for collection are provided on Table 4-1. Although a discussion of the sampling procedure is provided below, detailed surface water sampling guidelines are provided in method number 5620001.

For each of the sampling locations identified, the hydraulics of the water course will be determined prior to collecting the sample in order to obtain the most representative sample. After identifying the most representative sampling point, a grab sample will be obtained. This will include collecting a sample in a large container, with the sample aliquoted into the sample containers. Grab samples represent the quality of the water at a particular point and time.

This grab sampling method may lead to the loss of volatiles during the aliquoting process. However, it is felt that it is more important that the sample is representative of that point in time, rather than collecting samples in the individual containers over a period of time. In addition, care will be taken to prevent unnecessary agitation of the sample in order to minimize the loss of volatiles.

If it is necessary to wade into the water course to obtain a sample, the team member shall be careful not to disturb bottom sediments and shall enter the water course downstream of the sampling location. While in reality, it is difficult to prevent disturbance of the sediments, it is the responsibility of the team member to minimize the introduction of sediments into the sample. If necessary, the sampling technician shall wait for the sediments to settle before taking the sample. Where the water course is moving, a depth integrated grab sample shall be obtained. A depth integrated sample is collected by lowering an open container against the flow, to a depth just above bottom. The container is then turned into the flow and raised at a rate that allows it to just fill when it reaches the surface. The sample shall be collected in the middle of the stream.

Field water quality parameters will also be collected as a part of this program. These parameters include pH, conductivity, and temperature. Each of these procedures are discussed below. The SIPM procedures for the collection of field water quality data are provided in Table 6-3.

Temperature will be measured to an accuracy of 0.1° centigrade with a conventional mercury thermometer or thermister. Measurement of pH will be accomplished with a standard reference-type glass electrode pH meter accu-

rate to within 0.1 pH units. As with temperature, pH will be measured immediately upon extraction of the sample. The pH meter will be rinsed and subjected to a two-point calibration. For samples where acidic pH values exist (e.g., less than 7.0), the meter will be calibrated with buffers of pH 7.0 and 4.0; for basic samples, buffer calibration points will be 7.0 and 10.0. The electrode will be rinsed thoroughly with distilled water subsequent to calibration and prior to immersion in each sample.

TABLE 6-3

FIELD WATER QUALITY AND  
SURFACE WATER/LEACHATE SAMPLE COLLECTION PROCEDURES

Discipline	Procedure	Method Number
Field Water Quality	Procedure for Determination of Dissolved Oxygen, YSI Model 57 Dissolved Oxygen Meter	5617001
	Operation Procedure YSI Model 33 S-C-T Meter (Salinity, Conductivity, Temperature)	5617002
	Operation Procedure for HaakeBuchler pH Stick	5617003
	Surface Water Sampling Sampling Guidelines	5620001

Electrical conductance (specific conductivity) will be measured with a pre-calibrated portable Yellow Springs Instruments (YSI) salinity-conductivity-temperature (SCT) meter. Calibration will be accomplished in the laboratory, prior to field use, using a standard potassium chloride (KCl) solution. As with other meters, the SCT probe will be corrected to 25° centigrade. If the SCT meter is not a self-correcting model, correction will be by calculation with the appropriate conversion values.

A summary of the surface water/leachate sample collection procedure is as follows:

1. See Figure 3 for sample locations.
2. Evaluate water course for surface water samples and select location for sampling.
3. Stand adjacent to or wade into water course (from downstream) to selected sampling location at mid stream.
4. Collect depth-integrated grab sample by lowering the larger container to desired depth in water course and slowly raise to surface. Avoid collecting sediment in container. Aliquote the sample into the following sample containers:
  - a. Hazardous Substance List (HSL) analysis:
    - 4 x 1-liter amber jars - filled 7/8 full
    - Add 2 drops of 1:1 HCL to each of 2 x 40 milliliter VOA vials - fill without head space
    - 1 x 1-liter polyethylene bottle - preserve to pH  $\geq 12$  with NaOH (cyanide)
    - 1 x 1-liter polyethylene bottle - After sample collection, preserve with nitric acid to pH  $< 2$  (total metals).
5. Collect an additional sample and measure pH, conductivity, dissolved oxygen, and temperature.
6. Label samples.
7. Prepare all necessary duplicates/blanks per Section 6.7.
8. Pack and ship samples per Section 6.2, follow EPA Chain-of-Custody (Section 6.7.2), and CLP documentation (Section 6.7.3). Cool ice chest to 4°C.
9. Decontaminate field water quality instruments per Section 6.7.4.
10. Document all activities and measurements according to Section 6.7.1.

#### 6.4 GROUNDWATER SAMPLE COLLECTION

In order to determine the potential effect of the Ordot Landfill on the groundwater quality in the area, a sample will be collected from an unused domestic well located at Washington Junior High School. The location of

the well is provided in Figure 2. In addition, the appropriate procedural methods from the SIPM for the collection of a groundwater sample are provided in Table 6-4. A brief discussion of the procedure involved in collecting a groundwater sample is provided below.

The sampling protocols for the well will include the following pre-sampling procedure:

- o Check the well for above-ground damage.
- o Remove the well cap (a wrench may be needed).
- o Measure and record the depth to water from top of casing with an electric sounder and record the time of measurement.
- o Determine the total depth of the well, if possible.
- o Measure distance from top of casing from ground surface.
- o Determine the volume of water in the well (Volume =  $\pi r^2 h$ , where  $r$  = radius of well I.D.,  $h$  = height of water column).

TABLE 6-4  
GROUNDWATER SAMPLE COLLECTION PROCEDURES

Discipline	Procedure	Method Number
Field Water Quality	Procedure for Determining Temperature of Groundwater	5617004
Groundwater Sample Collection	Procedures for Water Level Measurement	5619007
	Procedure for Well Evacuation	5619008
	Considerations for Sample Withdrawal from Wells	5619009
	Procedure for Filtration of Samples	5617007

All of these procedures will be documented in the field logbook.

Following these preliminary procedures, the well should be purged prior to the collection of a groundwater sample. This process is a common procedure employed to assure the collection of a representative groundwater sample. To avoid the collection of the unrepresentative groundwater samples, the well is purged until three to ten well volumes are withdrawn. The well will be purged utilizing the pump presently installed in the well.

As previously discussed, three to ten well volumes should be removed. The specific number of well volumes should be based on field water quality data. For example, the specific conductance, pH and temperature of the discharge should be measured during the purging process. These parameters will provide a good indication of when representative aquifer water is being obtained. Therefore, when the conductivity and temperature have stabilized after the minimum amount of water has been withdrawn from the well (i.e., approximately three well volumes), a groundwater sample can be obtained. However, realizing that hydrogeologic conditions at the site are complicated, the purging process will be discontinued following the evacuation of ten well volumes, regardless of the stability of the conductivity and temperature of the discharge. This judgment will be performed by the on-site geologist or engineer. The purged water will be handled in accordance with the Guam Environmental Protection Agency (GEPA) regulations and procedures.

Following the pre-sampling purging processes, and with the exception of the VOA samples, the water level will be allowed to recover to the approximate pre-purging level and a representative groundwater sample will be collected. The VOA samples will be collected as soon as possible following the purging process and preserved with HCL. The samples will be collected using a teflon bailer with a disposable cable. The samples will be collected adjacent to the screened interval. If the well is inaccessible, then a sample will be collected directly from a discharge point, as close to the well head as possible.

As an integral component of the sampling program, a sample will be collected for field determinations of groundwater temperature, pH, and electrical conductance. Water level measurements will also be performed before and



after purging. The procedures for these field water quality determinations were provided in the previous section. The results of these determinations will be recorded in the field logbook.

The sample collected for dissolved metals analysis will include filtration through a 0.45 micron filter. The filtering of the sample for dissolved metals allows for preservation, since preservation of a non-filtered sample would result in higher dissolved metals than are present in the groundwater system. Following filtration, the sample will be preserved with nitric acid to a pH of less than 2. These procedures are in SIPM procedure numbers 5617007 and 5622006, which are provided in the Appendix.

The water level will be measured in the well, if it is accessible, during the sample collection effort. As previously mentioned, the water level during the pre-sampling casing evacuation will also be measured during the purging process to adequately document the complete withdrawal of the required casing volumes. An electric well sounder will be used to measure water levels.

A summary of the groundwater sample collection procedure is as follows:

1. Record pertinent well data from owner or operator to include:
  - o Date drilled, drilling method, bore diameter, and depth.
  - o Type of well casing, depth, diameter, perforated intervals.
  - o Type of pump, pump setting, discharges, water use.
2. Prior to starting pump, record static water level (Procedure No. 5619007).
3. Evacuate well by pumping (Procedure No. 5619008).
4. Record time evacuation began and names of participating field personnel.
5. Record volume of water evacuated using in-line meter, if available, 5-gallon bucket, 55-gallon drum, or portable v-notch weir, as appropriate.
6. Evacuate three to ten well volumes and measure field parameters (Procedure No. 5619007). Following stabilization of field parameters after evacuation of 3 well volumes or evacuation of 10 well volumes, collect required representative water samples.

7. From each well, the following samples will be collected directly from the well head:
  - o Sample for field analysis (pH, temperature, and EC):  
1 x 1-liter polyethylene bottle
  - o Samples for RAS-CLP laboratory:  
4 x 1-liter amber glass bottles (semi-volatiles, PCB's/pesticides)  
  
Add 2 drops of 1:1 HCL to each of 2 x 40 ml glass vials with teflon septa (volatiles) - fill without head space  
  
1 x 1-liter polyethylene bottle (cyanide)  
  
1 x 1-liter polyethylene bottle (metals)
  - o The duplicate sample for the well will be collected by simultaneously filling the sample container for each sample type. For example, the VOAs will be filled all at once for the sample and the duplicate, the same will occur for the semi-volatiles, etc.
8. Filter and preserve metals sample per EPA requirements (i.e., filter through .45 micron filter, preserve with nitric acid to pH <2).
9. Preserve cyanide sample with NaOH to pH >12. Cool to 4°C.
10. Label and pack samples per Section 6.2.
11. Log all samples in field book, including results of field analyses, time pumping stopped, and final pumping water level.
12. Follow EPA chain-of-custody procedures for sample shipment (Section 6.7.2).
13. Follow CLP Documentation Procedures with respect to Traffic Reports (Section 6.7.3).

#### 6.5 GEOLOGIC RECONNAISSANCE

One of the objectives of the ISC is to determine the composition of the bedrock material in the area directly surrounding the landfill. To achieve this objective, a bulldozer will be used to clear the vegetation and excavate trenches to a depth of approximately five feet for evaluation of the bedrock surface. The locations of the trenches are provided in Figure 3. The evaluation will take place in the field and will be noted in the site

logbook. Following the evaluation, the excavation will be reclaimed and restored to the natural state in accordance with GEPA guidelines.

Once a trench has been excavated, the soil material overlying the bedrock will be visually described and entered into the field logbook. It is anticipated that a weathered soil horizon will exist to a depth of approximately 3 to 4 feet. The detailed description of the soil material will include the following information:

- o horizonation
- o color utilizing Munsell Color notation
- o texture using USDA or Unified Soil Classification System
- o density or consistency
- o relative moisture content
- o visual inspection of relative porosity

The native bedrock material will also be determined at each of the proposed trench locations. The Ordot Landfill is situated south of the fault shown on Figure 2 and is presumably located on the volcanic Alutom Formation. This formation primarily consists of pyroclastics ranging in size from tuffaceous shale to coarse conglomerate and breccia. If the Alutom Formation is present under the landfill, then the potential for groundwater contamination is limited since the Alutom may be hydraulically separated from the water-bearing limestone unit located north of the fault. However, observations conducted during the field visit indicate that the landfill may be located on the Agana Member of the Mariana Limestone. If this is the case, there is a potential for hydraulic connection between the landfill and the sole-source aquifer. The Agana Member is generally yellow to tan in color and is characterized by a high content of calcareous mud. Due to the contrasting lithologies, the two formations will be easily distinguishable in the field. The geologic information which will be collected as a part of the visual inspection of the bedrock will include color, grain size, texture, mineralogy, sorting, etc. This information will be entered in the site logbook.

## 6.6 AIR QUALITY RECONNAISSANCE

The objective of the reconnaissance level air quality monitoring program is to provide qualitative data demonstrating the occurrence of emissions from the landfill. As previously described in Section 4.5, a more focused and detailed air quality monitoring program can be developed as a part of Phase II studies if significant emissions are detected. The approach to the study is presented below.

The following field instruments will be required in conducting the Phase I field program as a part of the Health and Safety Plan: a Century Portable Organic Vapor Analyzer (OVA), an HNu Photo-ionization Detector, a Ludlum Radiation Meter, and a Gastechtor Hydrocarbon Survey instrument. In that these instruments will be on-site for health and safety considerations, field personnel will also utilize the instruments to perform surveys along the boundary of the site to determine the presence of any emissions from the landfill. In addition, the landfill will be traversed in order to identify any emissions originating from specific locations in the landfill. The locations of the traverses will be based on field observations, accessibility, interviews with site workers, and discussions with local health and regulatory officials. The traverse locations and associated instrument responses will be noted in the field logbook. In addition, instrument responses observed along the boundary of the landfill will be noted. The operational procedures for the instruments are provided in the Appendix and are identified in Table 6-5.

Use of this suite of instruments will result in the detection of organic vapors and gases, including methane, emanating from the landfill. However, the following limitations are associated with the instruments:

- o In general, the detection limits for the instruments are on the order of 0.1 to 10 ppm, depending on the compound.
- o In very high humidity environments, condensation can collect on the HNu photo-ionization lamp, resulting in reduced instrument response.
- o The instruments do not readily provide for identification of detected gases or vapors, but can be used to screen for the

presence of methane. Methane is expected to be the main carrier gas at the landfill.

In regard to the detection limit for the instruments, organic concentrations less than 1 ppm detected at the landfill in an open environment are not expected to pose a threat to the public health and safety off-site. Therefore, until it is determined that the potential exists for emissions exceeding the 1 ppm level, a more elaborate monitoring program to detect and quantify the organic constituents at lower levels is not appropriate at this time. In addition, the potential problem associated with the HNu is easily corrected through routine cleaning of the lamp. The procedure for maintenance of the HNu is identified in Section 6.7 - Quality Assurance. The other instruments are not affected by high humidity environments.

TABLE 6-5

AIR SAMPLING QUALITY ASSURANCE PROCEDURES

Procedure	Method Number
Operation Procedure for HNu Model PI 101 Photoionization Analyzer	5607001
Operation Procedure for Century Portable Organic Vapor Analyzer (OVA), Model 128	5607003
Operation Procedure for AID Model 710 Hydro-Carbon Analyzer	5607002
Procedure for Operation of the CGA MINIRAM Particulate/Aerosol Monitor Model PDM-3	5607021

## 6.7 QUALITY ASSURANCE

This section describes field quality assurance activities for the Ordot Landfill site and complies with REM II requirements for accuracy, precision, representativeness and completeness for all activities. These requirements are consistent with those in the REM II Site Investigation Procedures Manual (SIPM). The SIPM procedures applicable to the Quality Assurance (QA) of this sampling effort are provided in Table 6-6. An overview of these procedures is provided below in the field investigations, chain-of-custody, Contract Laboratory Program (CLP), and the equipment sections.

### 6.7.1 Field Investigations

Field investigation conducted in support of the ISC will include the collection of three (3) surface water (including one (1) duplicate), eleven (11) leachate (including one (1) duplicate), two (2) groundwater samples (including one (1) duplicate), and three (3) travel blanks. The sample procedures were discussed previously in this section. Sample collection quality assurance activities are provided below.

Duplicate and Blank Sample Requirements - As a part of CDM's quality assurance program, duplicate and blank samples will be submitted to the laboratory for analysis. Details regarding these measures are as follows:

- o Duplicate Sample - One duplicate sample, per site, per day, per matrix is collected (or 10% of the total samples, per site, per day, per matrix per lab; whichever results in the greater number of duplicates). The actual sample location will be selected at the site based upon quantity and homogeneity of sample medium as well as from sample locations which are known or suspected to be contaminated, to obtain as true a duplicate as possible. The duplicate sample will be collected, numbered, packaged, and sealed in the identical manner as the other samples. In this way, the identity of the duplicate sample will be unknown to laboratory personnel performing the analysis. It is anticipated that the

TABLE 6-6  
GENERAL QUALITY ASSURANCE PROCEDURES

Procedure	Method Number
Chain-of-Custody	5622005
Procedure for Use and Maintenance of Field Notebooks	5621004
Equipment and Instrument Calibration and Maintenance - General	6600001
Calibration and Maintenance Procedure for Yellow Springs Instrument (YSI) Model 33 S-C-T Meter	6617002
Calibration and Maintenance Procedure for HaakeBuchler pH Stick	6617003
Calibration Procedure for the HNu PI 101	6607001
Calibration and Maintenance Procedures - Century Systems Portable Organic Vapor Analyzer - Model OVA-128	6607003

following duplicates will be collected: one groundwater sample from site GW-1, a surface water sample from site SW-2, and one from the eight leachate samples.

- o Blank Sample - Water samples which are being analyzed for volatile organic compounds will have a VOA "travel blank" in each shipping container. The blanks will be numbered, packaged, and sealed in the identical manner as the other water samples collected and will be sent blind to the laboratory. Certified organic-free water should be used to assemble water blanks. This water will be obtained from a certified source. The subsequent analysis of the blank water samples are a check against cross-contamination at collection, in transportation, and within the laboratory. It is anticipated that three to four water blanks will be sent to the laboratory.

Rinsate blanks will not be submitted to the laboratory for analysis. This is due to the fact that the use of sampling equipment will be limited to one-time use of the bailer and surface water/leachate sample container which will be decontaminated previous to field use. In addition, the leachate samples will be collected using the actual sample containers, thus avoiding the use of sampling equipment.

These blank, background, and duplicate requirements will result in an accurate characterization of field activities with respect to equipment decontamination, sampling representativeness, and sample handling.

Field Documentation and Logbooks - Field logbooks provide a means for documenting all data collecting activities performed at a site. As such, entries should be as descriptive and detailed as possible, so that a particular situation could be reconstructed without reliance on the collector's memory.

Field logbooks shall be bound and, preferably, contain water resistant paper with consecutively numbered pages. Logbooks shall be permanently assigned to field personnel, but are to be stored in the CDM Project Library when not in use. Each logbook is identified by the DCC number which indicates:

ZJX - FN - XXXX - XX

(Project Number - Field Notebook - Owner Identification - Sequence Number)

The cover of each logbook contains the following information:

- . Person or Organization to whom the book is assigned
- . Book number
- . Project name
- . Start date
- . End date



Entries into the logbook may contain a variety of information. At the beginning of each entry the following information is recorded: the date, start time, weather, all field personnel present, level of personal protection being used on-site, and the signature of the person making the entry should be noted.

All measurements made and samples collected are recorded. All entries should be made in pen. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialled. Entries should be organized into easily understandable tables, if possible.

Samples are to be collected following the procedures described in this sampling plan. The equipment used to collect samples should be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. In addition, the identification of the container number into which the sample is placed in the field is recorded. Sample numbers are assigned prior to going on-site and will be recorded in the field notebook, along with the corresponding Traffic Report numbers and CLP case numbers. Duplicates, which receive an entirely separate sample number, are noted under sample description. Significant field logbook entries (samples collected, significant observations) must be countersigned by another member of the project team.

#### 6.7.2 Sample Chain-of-Custody Requirements

Chain-of-custody procedures provides documentation of the handling of each sample from the time it is collected until it is destroyed. Such a written record is especially important if the results of analyses of samples will be used to support litigation.

To maintain a record of sample collection, transfer between personnel, shipment, and receipt by the laboratory which will analyze the sample (which will then continue the chain-of-custody within their laboratory records), a "Chain-of-Custody Record" is filled out for each sample type at each sampling location. Each time the samples are transferred to another

custodian, signatures of the person relinquishing the sample and receiving the sample, as well as the time and date, should document the transfer.

A serialized sample chain-of-custody record will also be derived from the U.S. EPA Region 9 Quality Assurance Management Section (QAMS). Actual field forms will include copies so that two forms are filled out simultaneously. The sampling team leader forwards a copy to EPA Region 9 QAMS, and the original remains with the samples until they are received by the laboratory. If samples are sent to different labs, a chain-of-custody will go to each lab. Care must be taken that all copies are legible. If additional duplicate sheets are required, the person relinquishing the samples is responsible for filling out additional copies, or making reproductions. A copy must be returned by the sampling team to the CDM Irvine office.

In addition to the chain-of-custody record, a Region 9 Sample Data Sheet will be completed for each sample collected. This data sheet is utilized to track samples on the regional level. This data sheet will be filled out in the field and returned to the Region 9 QAMS by the sample team leader at the end of the sampling effort.

The chain-of-custody record and other shipment records will be placed in a ziplock bag and taped to the lid of all shipping and transport containers prior to shipment. All samples will be shipped by commercial carrier or international carrier to the laboratory specified by the Sample Management Office (SMO). Samples should be packed so that no breakage will occur according to the protocols provided in Section 6.2. Samplers will notify the EPA Region 9 QAMS by telephone of the airbill number when samples are shipped.

The shipping or external container will be sealed with strapping tape and chain-of-custody seals and initialled so that any sign of tampering is easily visible. However, due to the potential for a customs inspection which will break the external seal, chain-of-custody seals will be placed on each sample.

### 6.7.3 Contract Laboratory Program (CLP) Documentation

The CLP will be utilized to perform the analytical services for the Ordot Landfill sampling program. The two analytical services available through the CLP include Routine Analytical Services (RAS), and Special Analytical Services (SAS). For the Ordot site, RAS services will be utilized for the water samples. To request the services, CDM will contact the U.S. EPA regional authorized requestor for the CLP, who can request the service from the SMO. The SMO will need to be informed of the sampling activity at least four weeks prior to scheduled start of sampling. In addition, the SMO requires the following information to initiate the RAS services:

- o Name(s), association, and telephone number(s) of sampling personnel.
- o Name and location of the site to be sampled.
- o Number and matrix of samples to be collected.
- o Type of analyses required (i.e., organics, inorganics).
- o Scheduled sample collection and shipment dates.
- o Nature of sampling event (i.e., ISC investigation).
- o Other pertinent information which may effect sample scheduling or shipment (i.e., anticipated delays due to site access, weather conditions, sampling equipment).
- o Name(s) of regional or contractor contacts for immediate problem resolution.

Once the request for the CLP services are processed, the SMO will issue the names and addresses of the laboratories performing the analysis and assign a case number to the sampling activity. A case number designates a single group of samples and is used to identify the sampling event throughout sample tracking and data production.

Once in the field, the sampling team will document and verify the collection of samples both in a field notebook and by using the appropriate traffic report forms. The sampling team leader will be responsible for completing SMO traffic reports, which are forms required to track the samples.

These forms will be provided by the U.S. EPA regional authorized requestor. In addition, details regarding procedures to be used to complete these forms are provided in the "User's Guide to the Contract Laboratory Program."

The following is a list of the traffic reports to be completed by the sampling team leader:

Organics Traffic Report - This form will be used to document the collection of water samples for organic analyses. Numbered blanks are filled in as follows:

1. Case number - sample site name/code - information provided by the SMO to the sampling team leader prior to sampling.
2. Sample concentration - check as appropriate.
3. Sample matrix - check as appropriate.
4. Ship to - provided by USEPA Region IX analytical coordinator.
5. Regional office - EPA Region 9, CDM, Irvine, California  
Sampling personnel: as appropriate  
Sampling date: as sampled
6. Containers and volumes - specify as appropriate. Be sure to mark volume level on sample bottle.
7. Name of carrier - commercial airline service unless otherwise specified. Airbill number: as shipped.
8. Sample description - check as appropriate.
9. Sample location - reference SAP sample number, field notebook reference and page number.
10. Special handling instructions - specify any special handling instructions. Also, specify the lot numbers for the sample containers provided by the bottle repository.

The preprinted and prenumbered adhesive sample labels affixed to the traffic report are transferred to the sample containers and referenced both in the sampler's field notebook and on the chain-of-custody form.

Inorganics Traffic Report - This form is used only to document the collection of water samples for inorganics analyses:

1. Case number-sample site name/code - all information is provided by the SMO to the sampling team leader prior to sampling.
2. Sample concentration - check as appropriate.
3. Sample matrix - check as appropriate.
4. Ship to - provided by USEPA Region IX analytical coordinator.
5. Regional office - EPA Region 9, CDM, Irvine, California  
Sampling personnel - as appropriate  
Sampling date - as sampled.
6. Shipping information name of carrier - Commercial airline service unless otherwise specified. Airbill number - as shipped.
7. Sample description - check as appropriate.
8. Volume level - check as appropriate - be sure to mark volume level on sample bottle.

The preprinted and prenumbered adhesive sample labels affixed to the traffic report shall be transferred to the appropriate sample containers and referenced in the sampler's field logbook and chain-of-custody form.

Sample Data Sheet - A Region 9 Data Sheet will be filled out for each sample in the following manner.

1. "Sample number" refers to the sample number in the upper right corner of the traffic report, e.g., MYA154, or the numbers on a SAS packing list in the sample numbers column, e.g., 1765Y-7.
2. "Case number" is a 4-digit number assigned by Sample Management Office (SMO) in chronological order.
3. "S.A.S number" is a 5-digit number ending in "Y", also assigned by SMO in chronological order, e.g., 1765Y. The Special Analytical Service (S.A.S) number should only be included (in item 3) on the sample data sheet when there is a special analytical service associated with that particular sample.
4. "Is this a "blank" sample?" If the sample is a blank or background sample, write Y and if it is not, write N.

5. "Matches sample number" refers to sample numbers of samples taken at the same station but sent for different analyses. If the sample listed after 1) on the sample data sheet is Y4543 (organic) it matches MY0045 (inorganic) and 1765Y-9 (a S.A.S. sample sent to another laboratory) only if these inorganic and S.A.S. samples are taken at the same location.
6. "Duplicate of sample number" differs from number 4, above, in that it refers to a duplicate (or split) sample taken at the same station, going to the same laboratory for the same analyses.
7. "Sample station" refers to the specific sampling location, e.g., MW 1 (monitoring well 1).
8. "Site name" should be standard through each sampling event at a given site. "City" and "State" are where the site is located.
9. "Site EPA ID number" is the ERRIS number, e.g., CAD005531099.
10. "Lead Sampler" use last name only. "Agency" refers to the agency or company the sampler works for.
11. "Project officer" is EPA project officer, last name only. "Section" refers to EPA project officer's mail code, e.g., T-2-2.
12. "Sample medium." See the back of the sample data sheet for encoding information.
13. "Contaminant concentration," low, medium or high.
14. "Statute under which sample was collected." All CLP samples are under CERCLA.
15. "Associated investigation ID number" is only to be filled out by EPA sampling personnel, all others may skip this line.
16. "Sample container type."
17. "No. of sample containers."
18. "Sample container(s) lot no." refers to the bottle lot number that is found on each bottle.
19. "Laboratory." A list of laboratory name abbreviations is printed on the back of the SAMPLE DATA SHEET.
20. "Analyses requested." When using the letter Z, write in the comments section (number 26) what Z equals, e.g., Z = low detection limits, Z = isopropanol.
22. "Chain of custody record no." is found in lower right corner of the Chain-of-Custody Record.

23. "Sample shipment airbill on.," a 9-digit number. If samples were not shipped by express service, i.e., hand delivered, indicate here.
24. "Date sample collected."
25. "Date sample sent to laboratory."
31. "Comment." In this column write what Z equals, indicate if the sample is a field blank or background, and any other comments you feel are pertinent.

The sample data sheet is to be filled out along with the rest of the paperwork at the time of sampling by the sampler and returned to EPA with the other paperwork.

If you have any questions, contact Stewart Simpson (415) 974-0925, or Laura Tom (415) 974-8595.

Distribution of Copies - The following is a form by form detail of copy distribution:

1. Chain-of-Custody Record - original accompanies samples, pink copy to QAMS, and a xerox copy to the sampler's files.
2. Both Inorganic and Organic Traffic Reports - original SMO, second copy (pink) to QAMS, third and fourth copies accompany samples and xerox copy for sampler's files.
3. Sample Data Sheet - send to QAMS.

QAMS address is:

U.S. EPA Region 9  
QAMS (P-3-2)  
215 Fremont St.  
San Francisco, CA 94105  
Attn: Stewart Simpson

SMO's address is:

U.S. EPA  
CLP Sample Management Office  
P. O. Box 818  
Alexandria, VA 22313

#### 6.7.4 Equipment Considerations

This section provides procedures for the maintenance and calibration, as well as the decontamination, of the field equipment. Maintenance schedules are provided in the SIPM procedures indicated in Table 6-3, which are presented in the calibration procedures are also provided as a part of these method numbers.

With respect to decontamination procedures, line and cables used to lower samplers into monitoring wells will be disposed of in an appropriate manner dictated by the on-site coordinator or decontaminated using the procedures outlined above for sampling devices. Persons obtaining samples will wear chemically inert protective gloves to prevent skin contact with sampling devices, lines and cables and potentially contaminated water.



## **Appendix A**

APPENDIX A  
SITE INVESTIGATION PROCEDURES

The following procedures, which were taken from CDM's "Site Investigation Procedures Manual," are provided in this Appendix.

<u>Procedure Title</u>	<u>Procedure Number</u>
Operation Procedure for HNu Model PI-101 Photo-ionization Analyzer	5607001
Operation Procedure for AID Model 710 Hydrocarbon Analyzer	5607002
Operating Procedure, Century Portable Organic Vapor Analyzer (OVA), Model 128	5607003
Procedure for Operation of the OCA Miniram Particulate Aerosol Monitor Model PDM-3	5607021
Procedure for Determination of Dissolved Oxygen, YSI Model 57 Dissolved Oxygen Meter	5617001
Operation Procedure YSI Model 33 S-C-T Meter (Salinity, Conductivity, Temperature)	5617002
Operation Procedure for Haakebuchler pH Stick	5617003
Procedure for Determining Temperature of Groundwater	5617004
Procedure for Filtration of Samples	5617007
Procedure for Water Level Measurement	5619007
Procedure for Well Evacuation	5619008
Considerations for Sample Withdrawal from Wells	5619009
Surface Water Sampling Guidelines	5620001
Procedure for Use and Maintenance of Field Notebooks	5621004

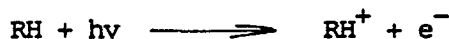
<u>Procedure Title</u>	<u>Procedure Number</u>
Sample Classification, Handling & Shipment	5622001
Sample Identification Procedure	5622002
Chain of Custody	5622005
Sample Bottle Preparation, Sample Preservation and Maximum Hold Times	5622006
Equipment and Instrument Calibration and Maintenance, General Requirements	6600001
Calibration Procedures for the HNu PI-101	6607001
Calibration and Maintenance Procedures Century Systems Portable Organic Vapor Analyzer Model OVA-128	6607003
Calibration and Maintenance Procedure YSI Model 33 S-C-T Meter	6617002
Calibration and Maintenance Procedures Haakebuchler pH Stick	6617003

OPERATION PROCEDURE FOR  
HNu MODEL PI 101  
PHOTOIONIZATION ANALYZER

1.0 INTRODUCTION

1.1 Operation Principle

The HNu Model 101 photoionization detector has been designed to measure the concentration of trace gases in many industrial or plant atmospheres. The instrument has similar capabilities outdoors. The analyzer employs the principle of photoionization for detection. This process is termed photoionization because the absorption of ultraviolet light (a photon) by a molecule leads to ionization via:



where RH = trace gas

$h\nu$  = a photon with an energy greater than or equal to an ionization potential of RH.

The sensor consists of a sealed ultraviolet light source that emits photons which are energetic enough to ionize many trace species (particularly organics), but do not ionize the major components of air such as O<sub>2</sub>, N<sub>2</sub>, CO, CO<sub>2</sub> or H<sub>2</sub>O. A chamber adjacent to the ultraviolet light source contains a pair of electrodes. When a positive potential is applied to one electrode, the field created drives any ions, formed by absorption of UV light, to the collector electrode where the current (proportional to concentration) is measured. The useful range of the instrument is from a fraction of a ppm to about 2,000 ppm.

## 1.2 Instrument Sensitivity and Calibration

The instrument responds to atmospheric compounds with ionization potentials equal to or less than the ionization energy of the UV light source. If a compound in air has an ionization potential greater than the energy source of the lamp, it will not be detected. Table 1 presents organic and inorganic compounds and the light sources that should be used to detect each compound. The instrument is capable of using 1 of 3 light sources - 9.5, 10.2, and 11.7 eV lamps. In addition, not all compounds respond equally to each light source and thus they vary in their sensitivity to ionization. As a result of varying sensitivities to photoionization, the response given by the instrument may or may not reflect the actual atmospheric concentration of the compound being detected. Reporting of instrument reading should thus be in terms relative to the calibration gas used, not in terms of actual atmospheric concentrations. Table 2 presents the relative sensitivities for various gases relative to a 10.2 eV light source. Use this table to determine the approximate response of the instrument to a compound of interest. The instrument can be calibrated to respond directly in ppm/volume by adjusting the response potentiometer to a known calibration gas for that compound. To do this, complete Procedure 6607001 and replace the isobutylene span gases with the compound of interest.

## 2.0 INSTRUMENT SPECIFICATIONS

### Performance

- o Range: 0.1 to 2,000 ppm
- o Detection limit: 0.1 ppm
- o Sensitivity (max) 0 to 2 ppm FSD over 100 division meter scale

TABLE 1 LAMP SOURCE IONIZATION POTENTIALS  
FOR ORGANIC AND INORGANIC AIRBORNE COMPOUNDS

9.5 eV Lamp Source	
Acids (organic)	Dimethyldisulfide
Alcohols	Dimethylsulfide
Amines	Ketones
Aniline	Phenol
Aromatics	Pyridine
Benzene	Styrene
Borontribromide	Toluene
Chlorinated aromatics	
10.2 eV Lamp Source	
Acetaldehyde	Chlorinated hydrocarbons
Acetic acid	Chloropenes
Acetone	Cyclohexanane
Acids (organic)	Dibromochloropropene
Acrolein (acetylates)	Dichloropropylene
Alcohols	Dimethyl disulfide
Aldehydes	Dimethyl formaldehyde
Aliphatics	Dimethyl sulfide
Alkyl halides	Epichlorohydrin
Allyl alcohol	Esters
Amides	Ethanol
Amines	Ethyl methacrylate
Ammonia	Ethylene
Aniline	Ethylene dibromide
Aromatics	Ethylene imine
Arsine	Ethylene oxide
Asphalt emissions	Furan
Benzene	Heterocyclics
Bromine	Hexane
Butane	Hexamethyl phosphoric triamide
Boron tribromide	Hydrazine
Carbon disulfide	Hydrogen sulfide
Chlorinated aromatics	Hydrogen selenide

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10.2 eV Lamp Source (Cont'd.)

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Iodine vapor	Phosphine
Isopropanol	Phosphorus trichloride
Ketones	Picolines
Lutidines	Pinene
Methyl bromide	Propylene
Methyl isocyanate	Pyridine
Methyl mercaptan	Pyrole
Methyl methacrylate	Styrene
Mineral spirits	Tetrahydrofuran
Naptha	Tetraethyl lead
Nitrates	Thionyl chloride
Nitrites	Toluene
Nitro alkanes	Vinyl acetate
Nitro benzene	Vinyl bromide
N-Octane	Vinyl chloride
Olefins	Vinylidene chloride
Phenol	
Phostoxin	

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11.7 Lamp Source

---

Acetic anhydride	Formic acid
Acetylene	Methanol
Acrylonitrile	Methylene chloride
Alcohols	Nitrates
Aldehydes	Nitrites
Alphatics	Nitro alkanes
Alkyl halides	Phostoxin
Butane	Propane
Carbon tetrachloride	Serafume
Chloroform	
Ethane	
Ethylene dichloride	
Formaldehyde	

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TABLE 2 RELATIVE SENSITIVITIES FOR VARIOUS GASES  
(10.2 eV Lamp)

Species	Photoionization Sensitivity*
p-xylene	11.4
m-xylene	11.2
benzene	10.0 (reference standard)
toluene	10.0
diethyl sulfide	10.0
diethyl amine	9.9
styrene	9.7
trichloroethylene	8.9
carbon disulfide	7.1
isobutylene	7.0
acetone	6.3
tetrahydrofuran	6.0
methyl ethyl ketone	5.7
methyl isobutyl ketone	5.7
cyclohexanone	5.1
naptha (85% aromatics)	5.0
vinyl chloride	5.0
methyl isocyanate	4.5
iodine	4.5
methyl mercaptan	4.3
dimethyl sulfide	4.3
allyl alcohol	4.2
propylene	4.0
mineral spirits	4.0
2,3-dichloropropene	4.0
cyclohexene	3.4
crotonaldehyde	3.1
acrolein	3.1
pyridine	3.0
hydrogen sulfide	2.8
ethylene dibromide	2.7
n-octane	2.5
acetaldehyde oxime	2.3
hexane	2.2
phosphine	2.0
heptane	1.7
allyl chloride (3-chloropropene)	1.5
ethylene	1.0
ethylene oxide	1.0
acetic anhydride	1.0
a-pinene	0.7
dibromochloropropane	0.7
epichlorohydrin	0.7
nitric oxide	0.6



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TABLE 2 RELATIVE SENSITIVITIES FOR VARIOUS GASES  
(10.2 eV Lamp) (Continued)

Species	Photoionization Sensitivity*
b-pinene	0.5
citral	0.5
ammonia	0.3
acetic acid	0.1
nitrogen dioxide	0.02
methane	0.0
acetylene	0.0

\*Expressed in ppm (v/v).

- o Repeatability:  $\pm 1$  percent of FSD
- o Linear range: 0.1 to 600 ppm
- o Useful range: 0.1 to 2,000 ppm
- o Response time: <3 seconds to 90 percent of full scale
- o Ambient humidity to 95% RH
- o Operating temperature: Ambient to 40°C (Instrument is temperature compensated so that a 20°C change in temperature corresponds to a change in reading of  $\pm 2$  percent full scale at maximum sensitivity).

#### Power Requirements and Operating Times

- o Continuous use on battery: approximately 10 hours
- o Continuous use with HNU recorder reduces instrument battery operating time to 1/2 normal time
- o Recharge time: maximum < 14 hours, 3 hours to 90 percent of full charge
- o Recharge current, maximum 0.4 amps at 15 VDC

### 3.0 INSTRUMENT CHECKOUT

3.1 Remove instrument box cover by pulling up on fasteners.

3.2 On the instrument panel there will be a label containing information on light source, calibration date, calibration gas, and span setting.

3.2.1 If the instrument has not been calibrated in the last 14 days or since its last field use, it should be recalibrated. Check the instrument status report form (F6265), which is maintained with the instrument in the instrument room, for its calibration history. If calibration is necessary, implement Procedure 6607001 before continuing with instrument checkout. For general

use the instrument should be calibrated to isobutylene at a span setting of 9.8.

- 3.2.2 Check the label for light source and refer to Table 1 for ionization potentials of various compounds. If the compound you wish to detect is not listed for the light sources provided with instrument, then the light source will have to be changed. Use the probe with the proper light source for the compounds to be detected.
- 3.2.3 Once it has been determined that the instrument has the correct lamp, the instrument may need to be recalibrated for the specific compound of interest. Use Procedure 6607001 to calibrate the instrument.
- 3.2.4 Check the battery supply by connecting the probe to the instrument box (see step 3.5), and turning the function switch to the battery check position (Figure 1). (Note: the battery check indicator will not function unless the probe is attached). The meter needle should deflect to the far right or above the green zone. If the needle is below or just within the green zone or the red LED indicator is on the battery should be recharged. Follow step 6.0 to recharge the battery.
- 3.2.5 Repack the instrument for shipment to the field.

#### 4.0 FIELD INSTRUMENT SET UP

- 4.1 Remove cover by pulling up on fasteners.

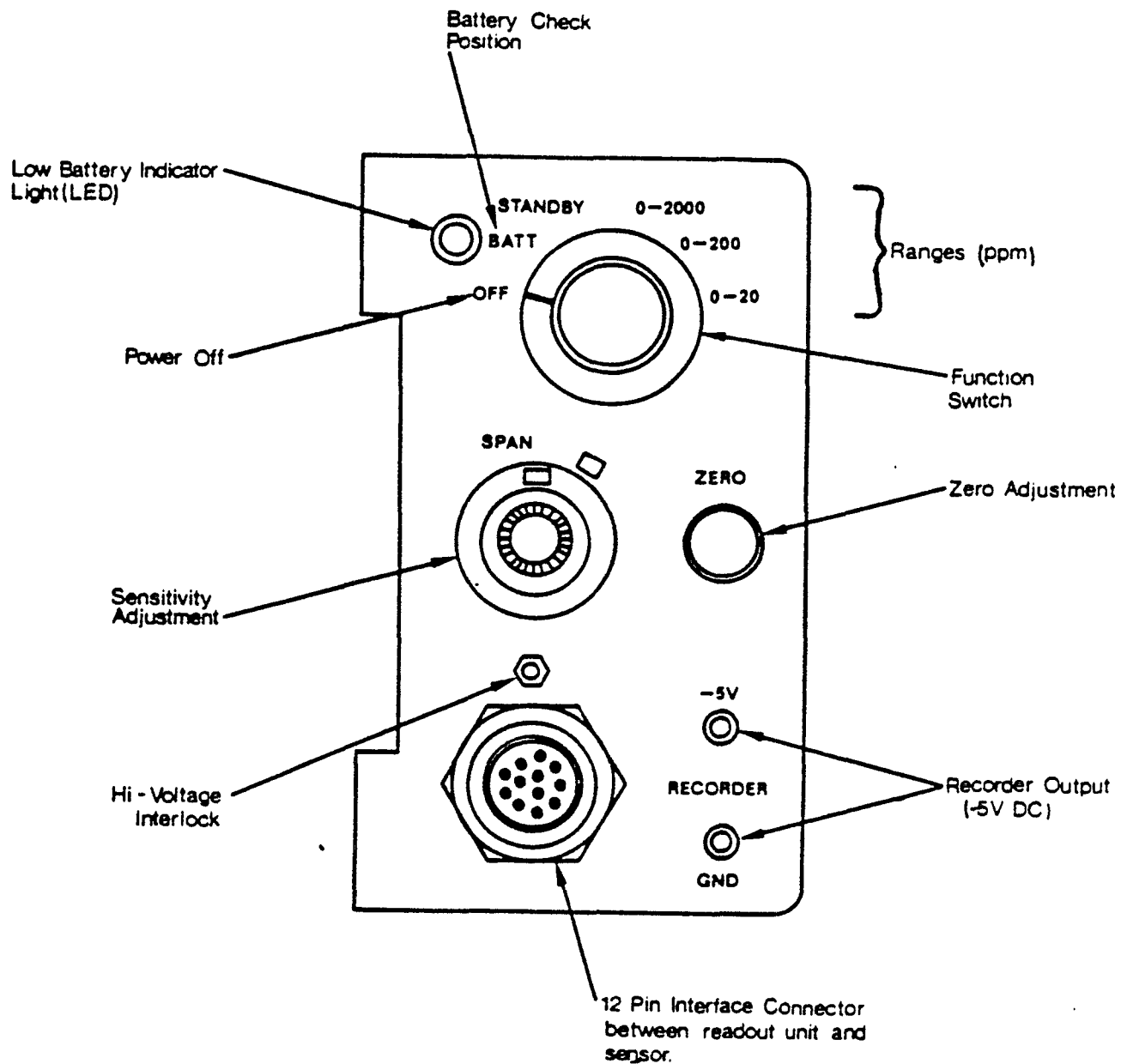


FIGURE 1 INSTRUMENT CONTROL PANEL FEATURES

- 4.2 Remove plate on cover to retrieve extension tube and carrying straps. Replace cover plate.
- 4.3 Check to see if instrument function switch is in the zero position before installing probe connector to instrument box. Turn instrument Off if found On.
- 4.4 Install 12 pin interface connector for the probe into the connector on the instrument box by carefully matching the alignment key of probe connector to the slots in the box connector. Twist the connector in a clockwise manner until a distinct snap and lock is felt.
- 4.5 Turn function switch to the battery check position. With an adequate charge, the needle on the meter should read within or above the green battery arc on the scale plate. If the needle is in the lower portion of the battery arc, the instrument should be recharged following steps 5.0 of this procedure. If the red LED comes on, the battery should be recharged.
- 4.6 To zero instrument turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotating yields a downscale deflection. No zero gas is needed because this is an electronic zero adjustment. Wait 15 to 20 seconds to ensure that the zero reading is stable. Adjust zero setting as necessary.
- 4.7 Attach probe extension into probe.
- 4.8 Turn instrument control switch onto 0-2,000 range position.

- 4.9 Gaze at end of probe opening for blue glow of UV light source. Avoid looking directly at lamp. Blue glow indicates photoionization source is working. The instrument should not be ready to measure atmospheric contaminants.

## 5.0 FIELD OPERATION

- 5.1 Place function switch in 0-20 ppm range for field monitoring. This will allow for the most sensitive, quick response in detecting airborne contaminants.
- 5.2 Before entering a contaminated area, determine background concentration. This concentration should be used as a reference to readings made in the contaminated area. Under no circumstance should one attempt to adjust the zero or span adjustments while the instrument is being operated in the field.
- 5.3 Take measurements in contaminated area, recording readings and locations. Should readings exceed the 0-20 scale, switch the function switch to the 0-200 or 0-2,000 range as appropriate to receive a direct reading. Return the instrument switch to the 0-20 range when readings are reduced to that level. Record measurements in notebook or on an appropriate form.
- 5.4 Keep in mind health and safety action guidelines for the level of protection you are wearing. Sustained readings above a certain level may force you to vacate an area or upgrade your level of protection.

Note: The instrument will not function properly in high humidity or when the window to the light housing is dirty. If the instrument response is erratic or lower than expected, implement Procedure 6607001.

5.5 When finished using the instrument reverse steps 4.1-4.5 above.

#### 6.0 INSTRUMENT RECHARGING

6.1 The instrument should be recharged 1 hour for each hour of use or overnight for a full day's use. (The battery will last 10 hours on a full charge).

6.2 To recharge the instrument:

6.2.1 Turn function switch to the off position.

6.2.2 Remove charger from instrument top compartment.

6.2.3 Place the charger plug into the jack on the left side of the instrument box.

6.2.4 Connect the charger unit to a 120V AC supply.

6.2.5 Check charger function by turning instrument switch to the battery check position. The meter should go upscale if the charger is working and is correctly inserted into the jack.

6.2.6 Place instrument in instrument mode and charge for the appropriate time period.

6.2.7 Turn instrument off following the recharge cycle.

## OPERATION PROCEDURE AID MODEL 710 HYDROCARBON ANALYZER

### 1.0 INTRODUCTION

#### 1.1 Operation Principle

The Model 710 portable total hydrocarbon analyzer is designed to measure organic vapor concentrations in an air sample. The instrument is a self contained unit operating from internal batteries and hydrogen gas supply. A pump within the sidepack pulls an air sample into the gun where the detector and hydrogen flame are located. Combustion of the sample in the hydrogen flame jet produces ions. These ions are in the presence of an electrical field due to the zero potential on the collector electrode and the bias voltage of the jet and are attracted to either the electrode or the jet. When the ions arrive at the electrodes, the ions either accept or release an electron. The electron transfer contributes to a small ion current which is measured by the electrometer amplifier connected to the collector electrode of the detector. The amplifier transmits the current through electronics which translate the current to a numeric reading on the digital readout. The electric current produced by the flame ionization is therefore converted via electronic circuitry into a part per million vapor in air reading when the instrument is correctly calibrated.

#### 1.2 Instrument Sensitivity and Calibration

Not all organic vapor molecules and compounds are ionized at the same rate using the flame ionization technique. Therefore, some molecules or compounds will show a greater or lesser response on the instrument readout based on the individual molecules response to flame ionization. Approximate response factors, relative to



methane, are shown in Table 1. The response factors are the values that the reading should be multiplied by to produce a correct reading for a compound other than methane. For example, when the instrument is calibrated to methane and one is measuring benzene, the response should be multiplied by 0.48 to obtain an approximate benzene in air measurement. Important. The response factors shown in Table 1 are only relative to methane (i.e., when the instrument is calibrated to methane) and the vapor being measured in air is known. When monitoring an unknown atmosphere, the instrument response can range from 10 percent to over 200 percent of the actual atmospheric concentration. This broad range of response to various atmospheric vapors is important to remember for health and safety considerations and when reporting monitored values.

It is possible to calibrate the instrument to read directly ppm in air vapors or gases other than methane. To do this implement Procedure 6607002 and as a calibration standard substitute the gas/vapor of interest for methane. When calibrated to a gas other than methane, response factors shown in Table 1 cannot be used. Contact the equipment manager to ascertain availability of span gases and the appropriate span gas to be used to calibrate the instrument for a compound of interest. For general work or working in unknown environments, the instrument will be calibrated to methane and the statements made in this section above will hold.

## 2.0 Specifications

Technique: Hydrogen flame ionization detector for organic hydrocarbons

Ranges: Digital readout  
0 to 200 ppm (resolution to 0.1 ppm)  
0 to 2,000 ppm (resolution to 1.0 ppm)

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TABLE 1  
APPROXIMATE RESPONSE FACTORS RELATIVE TO METHANE

Compound	Response Factor
Methane	1.00
Propane	1.30
Butane	1.04
Hexane	0.87
Benzene	0.48
Toluene	0.65
Ethanol	2.4
Isopropanol	0.75
Methyl Ethyl Ketone	0.90
Methyl Isobutyl Ketone	0.85
Chloroform	2
Carbon Tetrachloride	10
Trichloroethylene	1.3

Minimum Detectable: 0.1 ppm methane

System Response Time: 5 seconds (to 90%)

Noise: Less than 0.1 ppm

Sampling Rate: Approximately 1,500 ml/min.

Nominal Operating Condition: 20°C, 101.3 KPa, 2.0 ml/min.  
hydrogen and 115/230 volts

Operating Period: Portable: 8 hours nominal  
External supplies: Indefinite

Fully Portable: Self contained rechargeable battery pack and  
hydrogen supply

Hydrogen: Recharge requires 1,800 psi supply. Operation  
requires 80 to 1,800 psi @ 20 ml/min. CB grade or  
better

Recharging Time: 16 hours battery, 5 minutes hydrogen

Fully Portable: Self contained rechargeable battery pack and  
hydrogen

### 3.0 Checkout Procedure

#### 3.1 Instrument Description

The Model 710 consists of two major units connected by the umbilical cord (Figure 1). Some of the controls, most of the electrical circuits, the battery pack, the hydrogen cylinder, flow control system and pump are located in the side pack. The side pack is carried over the shoulder via the attached shoulder strap. The umbilical cord contains the gas line and the electrical connections between the side pack and the gun. The gun contains the flame ionization detector, the electrometer amplifier circuit, the digital readout and its circuitry, the sensitivity control and range switch, and the ignitor circuit and flame-out indicator.

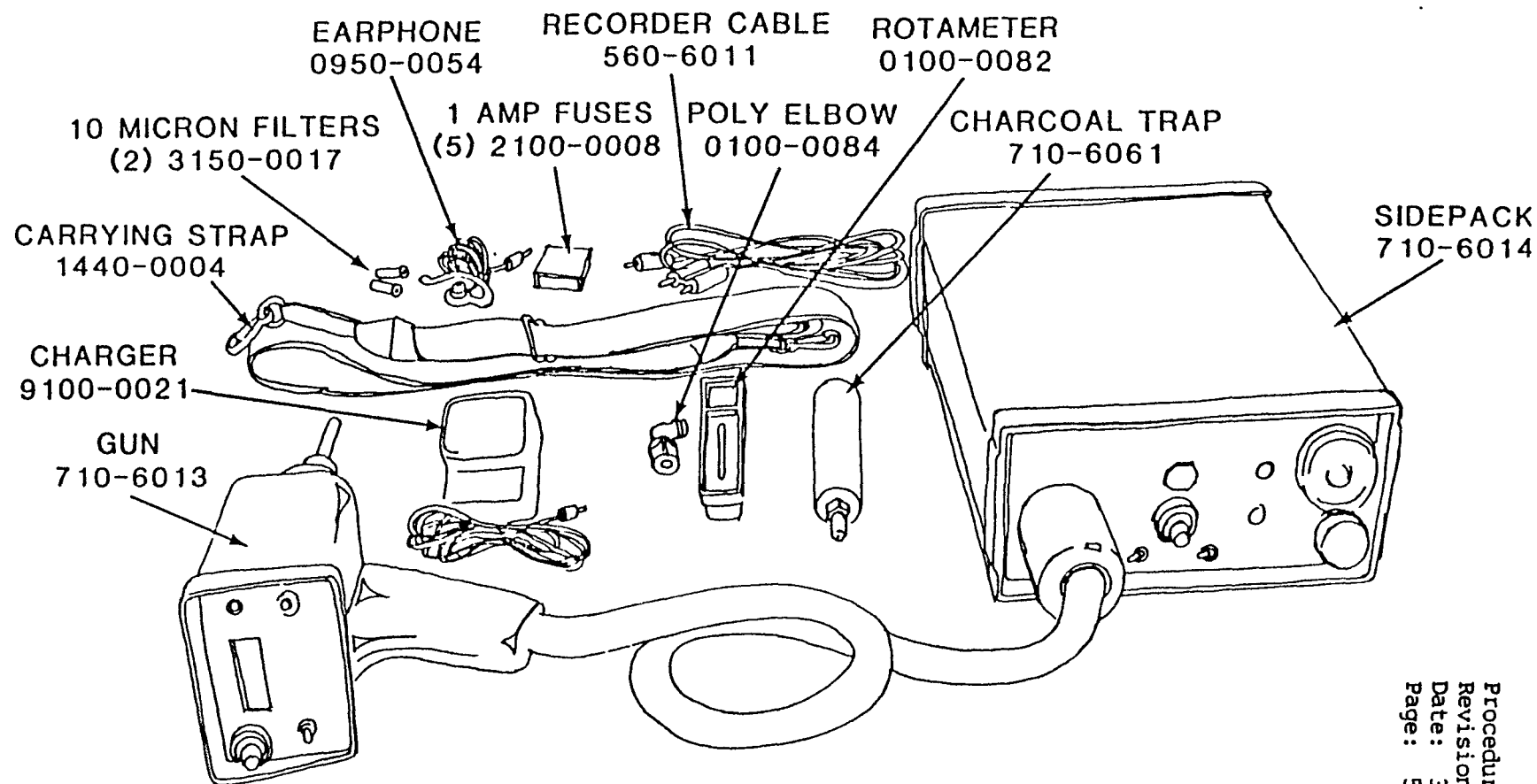


Figure 1

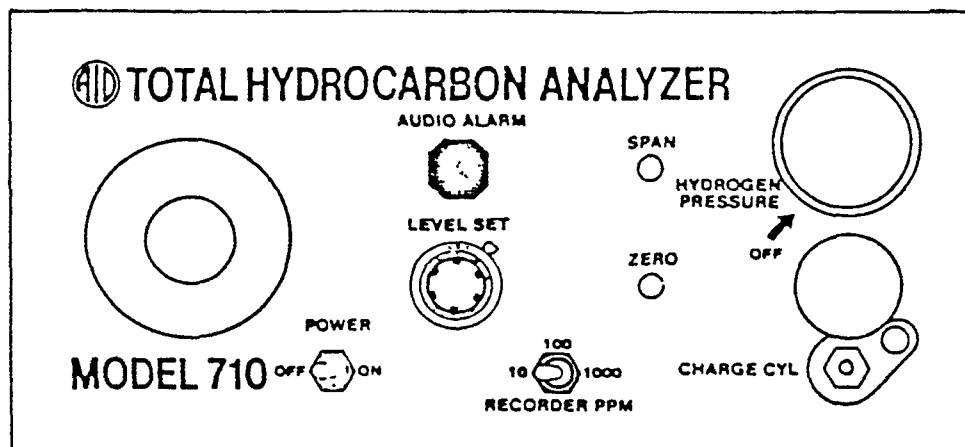
### 3.2 Instrument Checkout

- 3.2.1 Obtain copy of the instrument's status report form (F6265) and check to see the last dates of calibration and battery recharge. If the instrument either has not been recalibrated since the last field use, has not been calibrated in the past 14 calendar days, or needs to be calibrated to a specialty gas (refer to calibration tag on the instrument for calibration gas), implement Procedure 6607002 prior to completing instrument checkout. If the instrument was not recharged since the last field use or does not have a record of a recent (within 30 days) recharging, recharge the instrument following the steps listed below. If the record indicates a recent recharge, go to step 2.2.2.

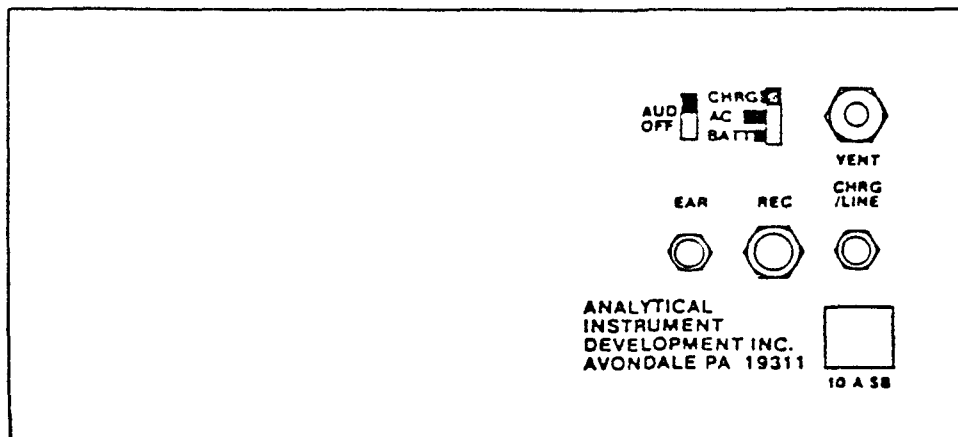
#### Battery Recharge:

1. Place the power switch on the rear panel of the side pack to the center or AC position.
2. Plug in the charger circuit to the charge line plug on the rear panel of the side pack (Figure 2). Caution anytime the charge plug is plugged into or removed from the instrument, the switch must be in the AC position. If the switch is in another position there is a possibility that the lamp fuse, located below the charge plug, will blow. This would then prevent operating the instrument from the battery or charging the battery.
3. Plug the charger into a 115 volt AC outlet.
4. Place power switch in the battery position.
5. Recharge instrument overnight (ca 14 hours).
6. Move power switch to the AC position.
7. Remove and unplug the recharger.

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FRONT



REAR

Figure 2 SIDEPACK PANELS

3.2.2 To check the battery, turn the power switch to the battery position and turn the instrument power switch on the front panel to the ON position. With adequate power from the battery, the pump is activated (i.e., is audibly apparent), the digital readout comes on (an erratic high or low value may be displayed), the red flame-out light next to the digital readout is on, and, if the alarm switch is ON the audible alarm will be heard. Check the alarm switch on the back of the instrument for the ON position. Place the switch in the ON position to ensure correct operation of this function. The flame-out alarm should go on with the switch in the ON position. (Note: This instrument does not have a low battery indicator. If one is not confident that the battery is completely charged, recharge the battery prior to field use.)

3.2.3 Next, check the hydrogen supply by opening the valve on the side pack. The pressure indicator should read 1,800 psi if the hydrogen supply tank is full. At 1,800 psi, the instrument will have approximately 8 hours of portable field use. If the gauge indicates less than 1,000 psi, the instrument will have 4 hours or less of field operation time. Therefore, the hydrogen supply tank should be refilled. If the tank has an adequate supply, continue to step 2.2.4.

To refill the tank:

1. Close hydrogen cylinder valve on side pack.
2. Remove the plug on the change cylinder port. (If the internal hydrogen supply lines are still charged, a small amount of gas may escape when the plug is removed).

3. Connect the 1/8 inch line from the hydrogen cylinder to the hydrogen cylinder charge plug, but do not tighten.
4. Momentarily turn on the regulator of the external hydrogen supply cylinder. Hydrogen should escape from the loose connection of Step 3. This purges the regulator in the connecting line. Three to five seconds is sufficient.
5. Turn off regulator (external supply).
6. Tighten fitting of Step 3.
7. Turn regulator of external hydrogen supply on to about 500 psi.
8. After this pressure has been on for one or two minutes (hydrogen has been flowing through the 710 flow system to purge the internal lines), open the hydrogen supply valve on the side pack.
9. Slowly increase the hydrogen pressure regulator to 1,800 pounds. The indicator for the hydrogen supply should show close to 1,800 pounds on the side pack. However, the gauge on the regulator, being a larger gauge, is the one that should be used to determine the 1,800 psi.
10. Close the hydrogen valve on the front panel of the instrument side pack.
11. Shut-off the high-pressure external hydrogen source.
12. Carefully loosen the tube fittings at the charge cylinder fitting on the side pack. There will be a small release of hydrogen as gas under pressure in the collecting lines escapes.
13. Remove fittings and hydrogen supply line from instrument.



14. Replace and tighten plug on the charge cylinder fitting on the instrument panel.

Note: Storage and use of hydrogen should meet all applicable standards.

#### 3.2.4 Instrument Function Check

1. Check instrument power switch on rear of side pack to ensure switch is in battery position. Place switch in battery position if not in this position.
2. Turn power switch on front of instrument panel to ON position. The pump motor should become audibly apparent, the flame-out alarm and LED should come on, and the instrument readout should come on. If this does not occur, repeat steps 3.2.1 and 3.2.2, or implement Calibration and Maintenance procedure 6607002.
3. Open valve for instrument hydrogen supply on side pack. Gauge should indicate pressure. Wait 2 minutes for hydrogen to purge instrument lines.
4. Press ignitor button on the gun. The pump will stop and in one to two seconds, a slight pop will be heard. This is the hydrogen igniting. Immediately release the ignitor button when the pop is heard. If the pop is not heard in 3 to 4 seconds, release button and wait 1 minute for the hydrogen to continue to purge the lines. If the flame does not ignite after repeated attempts to ignite, refer to

Calibration and Maintenance procedure 6607002 to troubleshoot the instrument.

5. When the ignitor button is released, the pump will restart and the flame-out light will go out when the hydrogen flame warms up the internal flame sensor (assuming the flame has been lit). If the flame-out light or alarm does not go out, repeat step 4. If the flame remains lit, the instrument display will slowly stabilize from an original high or off scale value. Wait 3 to 5 minutes for the instrument to stabilize.
6. Once the instrument has stabilized (i.e., display is showing stable readings), the instrument is now ready to be tested. Under no circumstances should the span, zero, or sensitivity adjustments be moved unless the instrument is being calibrated.
7. To ascertain whether the instrument is responding to hydrocarbons, pass the point of a "magic marker" pen, or similar device, in front of the probe. The instrument display should respond to the hydrocarbon source. If the instrument does not respond, use another hydrocarbon source to test instrument. If again no response is obtained, repeat steps 1 to 7 above or refer to Calibration and Maintenance Procedure 6607002 to troubleshoot the instrument.
8. Once it has been determined that the instrument is functioning properly, turn off hydrogen supply valve and the instrument power switch. The instrument is now ready for field use.

#### 4.0 FIELD OPERATIONS

4.1 The starting of the instrument is essentially the same as step 3.2.4 above. The steps are summarized below.

1. Turn power switch on the front of the instrument to the ON position. If the instrument does not come on, read step 2.0 above.
2. Open valve on instrument to initiate flow of hydrogen. Wait 2 minutes to charge instrument lines.
3. Ignite hydrogen flame by depressing ignitor button for 3 to 4 seconds or until a pop is heard. (If the instrument does not ignite, refer to 3.2.4 above).
4. Wait 3 to 5 minutes for instrument to stabilize.
5. If the instrument stabilizes at reading above 0, it may be necessary to zero the instrument using a zero span gas or the carbon filter supplied with the instrument. To calibrate with a zero span gas implement Procedure 6607002. To calibrate with the carbon filter, remove the probe from the gun and attach the filter to the gun. (The carbon filter will absorb all hydrocarbons except methane. Adjusting the zero potentiometer with the filter on will therefore produce a "zero" reading that does not include methane.) Adjust the zero potentiometer until the instrument reads zero. Remove the carbon filter and replace the probe. (If the instrument gives a reading above zero when the carbon filter is removed, that reading will be the background non-methane hydrocarbons.)

4.2 The instrument is now ready for field use. With a fully charged battery and a full hydrogen supply (1,800 psi) the instrument can be used for 8 hours of field use. Set alarm potentiometer on instrument to level at which the alarm is desired to be activated. When during the taking of field measurements hydrocarbon concentration at the alarm level is reached, the alarm will be activated.

4.3 Take and record background readings first. It may be necessary to subtract out background readings from contaminated zone readings. Record background readings on the appropriate form or log book.

- 4.4 Take measurements in contaminated zone area and record readings in appropriate form or log book. Extreme care and caution must be taken during field operations to ensure that the instrument does not come in contact with a contaminated materials. DO NOT CONTAMINATE THIS INSTRUMENT.
- 4.5 Keep in mind your health and safety action guidelines for the level of protection you are wearing. Sustained readings above a certain level may force you to vacate an area or upgrade your level of protection.
- 4.6 Following completion of field activities, the instrument is deactivated by turning off the hydrogen supply at the valve and turning off the power switch.
- 4.7 Unless the instrument is only being used for short periods (1-2 hours) it is best to recharge the battery and refill the hydrogen supply after each use. Follow steps 3.2.2 and 3.2.3 to recharge and refill the instrument.

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## OPERATING PROCEDURE CENTURY PORTABLE ORGANIC VAPOR ANALYZER (OVA), MODEL 128

### 1.0 INTRODUCTION

#### 1.1 Operating Features

The Century portable organic vapor analyzer (OVA) is designed to detect and measure gases and organic vapors in the atmosphere. The instrument utilizes the principle of hydrogen flame ionization for detection. The instrument measures gases and vapors by producing a response to an unknown sample, which can be related to a gas of known composition to which the instrument has been previously calibrated. During normal survey mode operation, a continuous sample is drawn into the probe and transmitted to the detector chamber by an internal pumping system. The sample flow rate is metered and passed through particle filters before reaching the detector chamber. Inside the detector chamber the sample is exposed to a hydrogen flame. When most organic vapors burn, they leave behind positively charged carbon containing ions which are collected by a negative collecting electrode in the chamber. An electric field exists between the conductors, surrounding the conductors, surrounding the flame and the collecting electrode. As the positive ions are collected, a current corresponding to the collection rate is generated on the input electrode. The current is measured with a linear electrometer preamplifier which has an output signal proportional to the ionization current. A signal conditioning amplifier is used to amplify the signal from the preamp and to condition it for subsequent meter or external recorder display. The meter display is an integral part of the Probe/Readout Assembly and has a scale from 0 to 10.

The instrument consists of two major assemblies: the Probe/Readout Assembly and the Side Pack Assembly. The output meter and alarm level adjustments are incorporated into the hand held Probe/Readout Assembly. The Side Pack Assembly contains the remaining operating controls and indicators.

## 1.2 Controls/Indicators

1. INSTR/BATT Test Switch - A three position toggle switch that in one position turns instrument on, except pump and alarm, in second position turns all power off, and third position displays battery charge condition on the readout meter.
2. PUMP (ON-OFF) Switch - This toggle switch turns on power to the internal pump and audio alarms.
3. Ignitor Switch - This momentary push button switch connects power to the ignitor coil in the detector chamber and simultaneously disconnects power to the pump.
4. CALIBRATE Switch - This three position toggle switch selects the desired range: X1 (0-10 ppm); X10 (0-100 ppm); X100 (0-1,000 ppm).
5. CALIBRATE ADJUST (Zero) knob - This potentiometer is used to "zero" the instrument.
6. GAS SELECT Knob (span control) - This ten-turn dial readout potentiometer sets the gain of the instrument commonly referred as span control.
7. Recharger Connector - This BNC connector is used to connect the battery pack to the battery recharger assembly.
8. H<sub>2</sub> TANK VALVE - This valve is used to supply or close off the fuel supply from the hydrogen tank.
9. H<sub>2</sub> TANK PRESSURE Indicator - This high pressure gauge measures the pressure in the hydrogen fuel tank which is an indication of fuel supply.
10. H<sub>2</sub> SUPPLY VALVE - This valve is used to supply or close off the hydrogen fuel to the detector chamber.

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11. H<sub>2</sub> SUPPLY PRESSURE Indicator - This low pressure gauge is used to monitor the hydrogen pressure at the capillary restrictor.
12. SAMPLE FLOW RATE Indicator - This indicator is used to monitor the sample flow rate.
13. Refill Connection - This 1/4 inch AN fitting is used to connect the hydrogen refill hose to the instrument.
14. REFILL VALVE - This valve is used to open one end of the instrument fuel tank for refilling with hydrogen.
15. Earphone Jack - This jack is used to connect the earphone; it turns off the speaker when in use.
16. VOLUME Knob - This potentiometer adjusts the volume of the internal speaker and earphone.
17. Readout and Sample Connectors - These connectors are used to connect the sample hose and umbilical cord from the Probe/Readout Assembly to the Side Pack Assembly.

## 2.0 Specifications

Sensitivity: 0.1 ppm (methane)

Response time: Less than 2 seconds

Readout: three ranges: 0 to 10 ppm, 0 to 100 ppm, 0 to 1,000 ppm, 250' linear scaled meter

Sample flow rate: Nominally 2 units

Fuel Supply: 75 cubic centimeter tank of pure hydrogen at maximum pressure of 2,300 PSIG, fillable while in case

Primary Electrical Power: Rechargeable and replaceable battery pack at 12 V DC

Service Life: Hydrogen supply and battery power - 8 hours minimum

Detection Alarm: Frequency modulated audible alarm. Can be preset to desired level. Frequency varies as a function of detection level.

Flame-out Indication: Audible alarm plus visual meter indication

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Battery Test: Battery charge condition indicated on the readout meter or battery recharger

Probe: Telescoping adjustment over 8 inches or probe can be completely removed from readout assembly.

Filtering: In line particle filters and activated charcoal filter

### 3.0 OPERATION PROCEDURE

Note: A condensed operating procedure checklist is provided inside the cover of the side pack.

#### 3.1 Instrument Setup

##### 3.1.1 Initial Assembly

Select Survey method (normal or "close area").

#### 1. Normal Survey Configuration:

- a. Connect the adjustable length probe to the Readout Assembly with the captive locking nut. Ensure that the probe is seated firmly in the Readout Assembly.
- b. Select the desired pickup fixture and check that a particle filter is installed.
- c. Connect the pickup fixture to the probe using the knurled locking nut.
- d. Connect the umbilical cord and sample hose to the Side Pack Assembly.

#### 2. "Close Area" Survey Configuration:

- a. Check to ensure that a particle filter is installed in the close area sampler.
- b. Connect the close area sampler directly to the Readout Assembly.
- c. Connect the umbilical cord and sample hose to the Side Pack Assembly.



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### 3.1.2 Fuel Supply

Check hydrogen supply by opening the H<sub>2</sub> Tank Valve and noting pressure. At least 1,600 pounds of pressure is required for 8 hours of usage. Refill tank if it contains less than 1,600 pounds and 8 hours of usage is required. The hydrogen supply tank is refilled following steps under 4.0 of this procedure.

### 3.1.3 Battery Check

Move INSTR/BATT test switch to the BATT position and note meter deflection on Readout Assembly. If the needle is below the battery mark on the readout, the battery will have to be recharged. Recharge battery following steps under 4.0 of this procedure.

### 3.1.4 Calibration

The instrument is normally calibrated to methane gas. When calibrated to methane gas, the GAS SELECT (span) Control is set to 300. Check the calibration label on instrument to determine date of calibration and calibration gas used. If the instrument requires recalibration, implement Procedure 6607003 before combining with this procedure.

## 3.2 Instrument Startup

The GAS SELECT control should be preset to the desired dial indication prior to turning instrument. This control is set during instrument calibration (Procedure 6607003) and must not be adjusted during instrument operation.

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1. Move Instrument (INSTR) Switch to ON and allow 5 minutes for warm up.
2. To set the audible alarm to a predetermined level (the level should be based on health and safety guideline, refer to the site health and safety plan for the action level):
  - a. Turn pump switch ON.
  - b. Adjust meter pointer to the desired alarm level using the CALIBRATE ADJUST (zero) knob.
  - c. Turn the Alarm Level Adjust knob on the back of the Readout Assembly until the audible alarm just comes on.
  - d. Adjust speaker volume with Volume Knob. If earphones are desired, adjust volume using earphones.
  - e. The instrument is now preset to activate the alarm when the level exceeds that of the setting.
3. Move the CALIBRATE Switch to X10 and adjust the meter reading to zero with the CALIBRATE ADJUST (zero) knob.
4. Check to see that the PUMP Switch is ON and observe the SAMPLE FLOW RATE Indicator. Indication should be approximately two units.
5. Open H<sub>2</sub> TANK VALVE one turn and observe reading.
6. Open H<sub>2</sub> SUPPLY VALVE one turn and observe reading. CAUTION: Do not leave H<sub>2</sub> SUPPLY VALVE open when the pump is not running. This will allow hydrogen to accumulate in the detection chamber.
7. Confirm that meter is still reading zero (readjust if necessary).
8. Depress ignitor button. Instrument pumps will go off. There will be a slight pop as hydrogen ignites. The meter pointer will go upscale of zero. Immediately after hearing the "pop", release ignitor button. Do not depress ingitor button for more than 6 seconds. If the hydrogen fails to ignite after 6 seconds, allow instrument to run for 2 minutes before attempting to ignite hydrogen again. If the hydrogen fails to ignite following repeated attempts at ignition, repeat entire instrument start up steps or refer to Procedure 6607003 for maintenance of the instrument.

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9. After ignition, the meter needle will indicate the background hydrocarbon level at area where instrument has been started. Note the background level in notebook or field log book.
10. Place CALIBRATE Switch in 1X range and move instrument to an area which is representative of the lowest ambient hydrocarbon concentration. Adjust the meter to 1 ppm with the CALIBRATE ADJUST (zero) knob. (Record all background readings.)

Note: The instrument has a low level (<0 ppm) alarm which indicates flame out. Placing or zeroing meter at 1 ppm will prevent normal background fluctuations from activating the low level alarm. This 1 ppm "zero" value must be subtracted from all measurements.

11. The instrument is now ready for field use.

### 3.3 Operating Procedures

1. Set the CALIBRATE switch to the 1X level. If, during taking measurements, the meter goes off scale, adjust CALIBRATE switch to 10X or 100X to record levels.
2. Take measurements at ground, waist, and head levels at all areas of the site to be surveyed. Drum openings, enclosures, etc. should be surveyed for organic vapors. Record all measurements in field log book or notebook. Record measurements relative to calibration standard, not as ppm in air.
3. When organic vapors are detected, the meter pointer will move upscale and the audible alarm will be activated if the level exceeds the preset value. The frequency of the alarm will increase as the level of exceedence is increased.
4. If the flame out alarm is actuated, ensure that the pump is running and depress the ignitor button. This should reignite the hydrogen. Flame out can be caused by explosive or oxygen deficient atmosphere. It can also be caused by constriction or blockage of the inlet tube.

### 3.4 Shut Down Procedure

1. Close H<sub>2</sub> SUPPLY VALVE.
2. Close H<sub>2</sub> TANK VALVE.

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3. Move INSTR Switch to OFF.
4. Wait 5 seconds and move PUMP Switch to OFF. The instrument is now in shut down configuration.

#### 4.0 FUEL REFILLING

The instrument should be completely shut down for hydrogen tank refilling. The refilling should be done in a ventilated area. There should be no potential ignitors or flame in the area.

1. Attach filling hose to external source of hydrogen. Pure hydrogen of 1,600 to 2,300 psi will be required.
2. Crack open valve on hydrogen supply, place FILL/BLEED valve on hose in FILL position and purge hose for 2-3 seconds.
3. Close FILL/BLEED Valve (OFF position) and immediately attach other end of fill hose to instrument fill connection and tighten the connection.
4. Open supply valve external on hydrogen tank 1/2 to 1 turn. Set regulator to 1,600 to 2,300 psi.
5. Open the REFILL Valve and the H<sub>2</sub> TANK VALVE on the instrument.
6. Place FILL/BLEED Valve in FILL position. The instrument hydrogen tank should now be filling.
7. The instrument H<sub>2</sub> Pressure Indicator should now indicate instrument tank pressure. This pressure should approximate that of the external supply tank regulator gauge.
8. After the instrument tank is filled, shut off:
  - a. The REFILL VALVE on the instrument panel.
  - b. The FILL/BLEED Valve on the filling hose assembly.
  - c. The valve on the external hydrogen supply bottle.
9. The supply hose and internal lines on the instrument now contain hydrogen under pressure. To reduce this pressure to atmospheric pressure:
  - a. Turn FILL/BLEED Valve to BLEED position until gas can no longer be heard escaping.

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- b. Turn FILL/BLEED Valve to FILL position to allow gas trapped in the connective fittings to go into the hose assembly.
  - c. Turn FILL/BLEED Valve to BLEED position to bleed off this pressure.
  - d. Turn FILL/BLEED Valve to OFF position. Keep valve in OFF position.
- 10. Close H<sub>2</sub> Tank Valve.
  - 11. Remove fill hose from instrument. Any gas still under pressure will escape as the connector is removed. Release of gas should only be momentary.
  - 12. As a check of the integrity of the instrument's hydrogen supply system, observe the H<sub>2</sub> TANK PRESSURE Indicator with the system shut down. Release of internal pressure should not go down rapidly. If there is a rapid decrease (greater than 350 PSIG/hour) with the instrument in shut down mode, there is a significant leak in the H<sub>2</sub> supply system. If so, the instrument should be returned to the manufacturer for repairs.

#### 5.0 BATTERY RECHARGING

The battery should be recharged following each use of 4 hours or more, or when the battery check indicator indicates need of a charge. Never charge instrument in hazardous environment or when refilling hydrogen tank.

- 1. Remove cover from battery charge part on instrument.
- 2. Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet.
- 3. Move battery charger switch to the ON position. The light above the switch should illuminate.
- 4. Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right while charging. When fully charged, the pointer will be in line with the charged mark above the scale.

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5. Approximately 1 hour of charging time is required for each hour of operation; 12 hours for complete charge. Do not charge for more than 24 hours.

#### 6.0 REFERENCE

Foxboro Analytical. No date. Instruction and Service Manual, Century Systems Portable Organic Vapor Analyzer Model OVA-128 M1 2R900AC.

PROCEDURE FOR OPERATION OF THE GCA MINIRAM  
PARTICULATE/AEROSOL MONITOR MODEL PDM-3

1.0 INTRODUCTION

The MINIRAM (Minature Real-time Aerosol Monitor) Model PDM-3 is an ultra-compact personal-size airborne particulate monitor whose operating principle is based on the detection of scattered electromagnetic radiation in the near infrared range. The MINIRAM is a light scattering aerosol monitor of the nephelometric type that continuously senses the combined scattering from particles present within its sensing volume (approximately  $1 \text{ cm}^3$ ). Air surrounding the MINIRAM passes freely through the open aerosol sensing chamber as a result of air transport caused by convection, circulation, ventilation, and personnel motion. Its scattering sensing parameters have been designed for preferential response to the particle size range of 0.1 to 10 micrometers. The MINIRAM measures the concentration of any airborne particles, both solid and liquid, and the display indicates this level in milligrams per cubic meter ( $\text{mg}/\text{cm}^3$ ), based on its factory calibration using a standard test dust. The MINIRAM can be used to measure the concentration of all forms of aerosol: dusts, fumes, smokes, fogs, etc.

2.0 SPECIFICATIONS

- o Measurement ranges:  $0.01$  to  $10 \text{ mg}/\text{m}^3$  and  $0.1$  to  $100 \text{ mg}/\text{m}^3$
- o Precision and stability (for 10 sec. readings at  $25^\circ\text{C}$ ):  
 $\pm 0.03 \text{ mg}/\text{m}^3$  (2 sigma)
- o Precision and stability of time-averaged measurements (at  $25^\circ\text{C}$ ):  
 $\pm 0.02 \text{ mg}/\text{m}^3$  (for 1 minute averaging)

- $\pm 0.006 \text{ mg/m}^3$  (for 10 minute averaging)
- $\pm 0.003 \text{ mg/m}^3$  (for 1 hour averaging)
- $\pm 0.001 \text{ mg/m}^3$  (for 8 hour averaging)
- o Temperature coefficient:  $0.005 \text{ mg/m}^3$  per  $^{\circ}\text{C}$  (typical)
- o Readout resolution:  $0.02 \text{ mg/m}^3$  or  $0.1 \text{ mg/m}^3$
- o Digital readout updating time: 10 seconds
- o Analog output time constant: 0.2 seconds
- o Total measurement period: 8 1/3 hours
- o Particle size range of maximum response 0.1 to 10  $\mu\text{m}$  in diameter
- o Measurement display: normally 10 second real-time measurement
- o Data storage: 7 concentration average, sampling period in minutes, off-time identification number, zero value, programmable code, and check sum
- o Real time outputs analog and digital ASCII
- o Memory playback: LCD display or ASCII digital output
- o Normal battery voltage: 7.5V
- o Average battery current drain 40 mA
- o Continuous operating time with full battery charge: 10 hours (approx.)

### 3.0 OPERATING INSTRUMENT

#### 3.1 Instrument Set Up

Assuming adequate battery charge (see Section 5.0), the instrument display should indicate one of the following:

1. Blank display - the instrument has been in off mode for more than 48 hours.
2. "OFF" display - the instrument has been in off mode for less than 48 hours.



3. Concentration (numeric) display - the MINIRAM is in instrument mode.

To start measurement cycle:

1. If instrument shows blank display, press OFF and wait until the display reads OFF before pressing MEAS to initiate measurement cycle.
2. If instrument shows OFF, press MEAS directly to initiate measurement cycle.

### 3.2 MEAS Function (Measurement)

Start monitoring operations by pressing MEAS key. The first readout is "GO", followed by last reading or 0.0. After approximately 36 seconds the first new 10-second-averaged concentration reading is displayed. All subsequent readings are concentration values in  $\text{mg}/\text{m}^3$ , updated every 10 seconds. The MINIRAM will now run in the measurement mode for 500 minutes (8 hours and 20 minutes). Once the MEAS mode has been entered, the mode can only be interrupted by pressing the OFF key. The instrument normally operates in the 0.00 to  $9.99 \text{ mg}/\text{m}^3$  range. When a 10-second concentration exceeds  $9.99 \text{ mg}/\text{m}^3$  the display automatically switches to the 0.0 to  $99.9 \text{ mg}/\text{m}^3$  range.

### 3.3 MEAS and TIME

If both MEAS and TIME are pressed at the same time, the instrument will operate continuously and will not shut down after 8.3 hours.

#### 3.4 OFF

When this key is pressed, the instrument will discontinue whatever mode it is in and shut down to a minimum power level (blank screen) within 10 minutes to 48 hours of depressing the key.

#### 3.5 TIME

During the measurement mode, if time is depressed, the display will show the elapsed time from the start of the measurement run. The instrument will return to the measurement mode when the key is released.

#### 3.6 TWA (Time Weighted Average)

If the TWA key is depressed during the measurement mode, the display will indicate the TWA concentration in  $\text{mg}/\text{m}^3$  since the start of the run.

#### 3.7 SA (Shift-Average)

Depressing the SA key during the measurement mode will provide an 8-hour TWA concentration.

#### 3.8 PBK (Playback)

With the instrument in the OFF mode, stored information can be played back by depressing the PBK. Depressing the key for more than 1 second will prompt the display to indicate day. First, the ID number is displayed; second, the run number (7 through 1);

third sampling time in minutes; fourth, off time between runs; and fifth, average in  $\text{mg}/\text{m}^3$ . By depressing the PBK key for less than 1 second, stored data will be fed out for printout, etc.

### 3.9 ZERO

A background level or zero value is automatically subtracted from all aerosol concentration readings during the measurement mode. Pressing ZERO during a measurement period provides a momentary display of the stored zero concentration. To update this value, the MINIRAM must be in OFF mode. Then press ZERO and wait until the display again indicates "OFF". The average of 4 consecutive 10-second zero level measurements will then be stored by the MINIRAM as the new ZERO reference value. The zero update should be performed in a clean air or background relatively dust-free environment; otherwise an elevated ZERO value may be entered into the instrument. The zero check on the instrument should be performed prior to each field event.

### 3.10 ID

Pressing the ID key will display the instrument identification number. This number can be changed by setting the instrument into program mode. (See instruction manual for a description of the use of the program mode.)

## 4.0 FIELD OPERATIONS

1. Determine background value by establishing the ZERO reference value following Section 3.9. Record background value in field notebook.

2. When monitoring particulates outdoors, place monitor in stationary area within the breathing zone, and shielded from the sun or bright or reflecting lights. Excessive variable illumination of the scattering detector can result in significant measurement errors.
3. An approximate vertical position is preferred for any long-term monitoring. Ensure that the detection chamber is not blocked to air flow in the vertical position.
4. Check and record the instantaneous and time weighted average readings at the frequency specified in the health and safety plan for the site.

#### 5.0 BATTERY RECHARGE

The battery should be recharged after each 8 hours of portable use. Minimum charge time is 12 hours. To charge the instrument:

1. Plug charger into A.C. line
2. Connect charger plug into corresponding MINIRAM receptacle
3. Leave charger connected for a minimum of 12 hours

Note: The BAT indicator bar on the LCD will display at any time the battery voltage becomes deficient.

#### 6.0 REFERENCE

GCA Corporation. 1984. MINIRAM Personal Monitor Model PDM-3  
Operations Manual.

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## PROCEDURE FOR DETERMINATION OF DISSOLVED OXYGEN, YSI MODEL 57 DISSOLVED OXYGEN METER

### 1.0 INTRODUCTION

The YSI Model 57 Dissolved Oxygen Meter is intended for dissolved oxygen and temperature measurement in water and wastewater applications, but is also suitable for use in certain other liquids. Dissolved oxygen is indicated in mg/l on 0-5, 0-10, and 0-20 mg/l scales. Temperature is indicated in 0°C on a -5°C to 45°C scale. The dissolved oxygen ranges are automatically temperature compensated for solubility of oxygen in water and permeability of the probe membrane, and manually salinity compensated.

The probe has Clark-type, membrane-covered polarographic sensors with built in thermistors for temperature measurement and compensation. A thin permeable membrane stretched over the sensor isolates the sensor elements from the environment, but allows oxygen and other gases to enter. When a polarizing voltage is applied across the sensor, oxygen that has passed through the membrane reacts at the cathode, causing a current to flow. The membrane passes current at a rate proportional to the pressure difference across it. Since oxygen is rapidly consumed at the cathode, it can be assumed that oxygen pressure inside the membrane is zero. Hence, the force causing the oxygen to diffuse through the membrane is proportional to the absolute pressure of oxygen outside the membrane. If oxygen pressure increases, more oxygen diffuses through the membrane and more current flows through the sensor. A lower pressure results in less current. The current is registered on the O<sub>2</sub> meter to reflect dissolved O<sub>2</sub> levels.

### 2.0 SPECIFICATIONS

#### 2.1 Instrument

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1. Oxygen Measurement  
Ranges: 0-5, 0-10, and 0-20 mg/l  
Accuracy:  $\pm 1\%$  of full scale at calibration temperature  
Readability: 0.025 mg/l on 0.5 scale; 0.05 mg/l on 0-10 scale; 0.1 mg/l on 0-20 scale.
2. Temperature Measurement  
Range:  $-5^{\circ}\text{C}$  to  $+45^{\circ}\text{C}$   
Accuracy:  $\pm 0.5^{\circ}\text{C}$  plus probe which is  $\pm 0.1^{\circ}\text{C}$   
Readability:  $0.25^{\circ}\text{C}$
3. Temperature Compensation  
 $\pm 1\%$  of D.O. reading for measurements made within  $\pm 5^{\circ}\text{C}$  of calibration temperature.  
 $\pm 3\%$  of D.O. reading over entire range of  $-5$  to  $45^{\circ}\text{C}$  probe temperature.
4. System Response Time  
90% in 10 seconds at constant temperature of  $30^{\circ}\text{C}$  with YSI 5775 membrane.
5. Operating Temperature Range  
 $-5^{\circ}$  to  $+45^{\circ}\text{C}$ . Large ambient temperature changes will result in 2% loss of accuracy unless red line and zero are reset.
6. Recorder Output  
0 to 114 - 136 mV. Recorder should have 50,000 ohms minimum input impedance.
7. Power Supply  
Two disposable "C" size carbon zinc batteries (Eveready 935C or equal) providing approximately 1,000 hours of operation.

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## 2.2 Probe

1. Cathode: gold
2. Anode: silver
3. Membrane: 0.001 inch teflon
4. Electrolyte: half saturated KCl
5. Temperature Compensation (see instrument specification)
6. Pressure compensation: Effective 0.5% of reading with pressure to 100 psi (230 feet of seawater)
7. Polarizing Voltage: 0.8 volts nominal
8. Probe current: Air at 30°C = 19 microamps nominal  
Nitrogen at 30°C = 0.15 microamps or less

## 3.0 OPERATING PROCEDURES

### 3.1 Required Equipment

YSI Model 57 Oxygen Meter  
YSI5740 detachable cable  
YSI5739 probe  
Clean cloth  
Distilled water

### 3.2 Preparation of the Probe

A YSI 5739 D.O. Probe is used with the instrument. All probes are shipped dry from the factory and must be filled with electrolyte prior to operation. To fill or replace the electrolyte, maintenance steps 2.4 in Procedure 6617001 should be followed.

If the probe is not connected to the cable, it can be reconnected using the following steps:

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1. Inspect the connector and O ring for cleanliness and condition. Clean if dirty, replace O ring if frayed or damaged.
2. Push the electrical connector into the probe body, rotating it until the two halves mate. A light coating of vaseline or silicone grease on the "O" ring will make reassembly easier. Air trapped between the connector halves may cause them to spring apart slightly; this is normal.
3. Hand tighten the retaining nut.  
Note: the connectors are not intended for frequent disconnecting. Therefore, it is best to keep the probe connected to the cable.

### 3.3 Preparing the Instrument

- 3.3.1 The instrument must be placed in its intended operating position - vertical, tilted back, or on its back - before it is prepared for use and calibrated. Readjustments may be necessary when the instrument operating position is changed.

#### 3.3.2 To Prepare the Instruments

1. With the switch in the off position, adjust the meter pointer to zero with the screw in the center of the meter panel. Readjustments may be necessary if the instrument position is changed.
2. Move switch to RED LINE and adjust RED LINE knob until the meter aligns with the red mark at 31°C position. If the needle will not reach the red line, the battery will have to be replaced.
3. Move switch to zero and adjust zero knob until meter aligns with zero mark.
4. Attach the prepared probe cable to the probe connector on the instrument body and adjust the retaining ring finger tight.



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5. Before calibrating, allow 15 minutes for optimum probe stabilization. Repolarize (steps 1 to 4) whenever the instrument has been off or the probe has been disconnected.

### 3.4 Field Calibration

#### 3.4.1 Calibration Methods

Three calibrations methods are available to calibrate the instrument: Winkler Titration, Saturated Water, and Air. However, because Winkler Titration and Saturated Water calibration require either calibration items or calibration times that may be prohibitive for hazardous waste site conditions, only the Air method is represented here. A description of the Winkler Titration calibration methods are presented in calibration and maintenance Procedure 6617001.

#### 3.4.2 Air Calibration

1. Place probe in moist air. This is done by wrapping the probe loosely with a damp cloth. Care must be taken so that the cloth does not touch the membrane. Wait approximately 10 minutes for temperature stabilization.
2. Adjust knob to TEMPERATURE and read valve. Refer to Table 1 and determine calibration value.
3. Determine altitude or atmospheric correction factor from Table 2.
4. Multiply the calibration value from Table 1 by the connection factor from Table 2. This will provide a corrected calibration value.
5. Adjust the main knob to the appropriate setting for the converted calibration value and set the Salinity knob to 0.

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6. Adjust the CALIBRATE knob until the meter reads the corrected calibration value from step 4.
7. Wait two minutes to verify calibration stability. Readjust as necessary.

The probe is now calibrated and should hold the calibration for many measurements. Calibration can be disturbed by physical shock, touching the membrane, or drying out of electrolyte. It is best to pre-check calibration after each series of measurements.

### 3.5 Dissolved Oxygen Measurement

1. With instrument prepared for use and the probe calibrated, place the probe into the sample to be measured.
2. If sample is not already being physically stirred (i.e., running water), stir sample by moving probe up and down in the liquid or container at about 1 foot per second rate.
3. Adjust salinity knob to the salinity of the sample. (If salinity is unknown, it will have to be determined with a conductivity meter).
4. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
5. Select meter setting to reflect range of D.O. concentration. Read dissolved oxygen level on meter and record on appropriate form or in log book.
6. When finished taking D.O. measurements, turn instrument off. Check sponge or paper in probe bottle cover for dampness. Dampen sponge or paper with distilled water if dry. Place bottle cover over probe to keep electrolyte from evaporating.

### 4.0 REFERENCE

Yellow Springs Instrument Company, 1983. Instruction Manual YSI Model 57 Dissolved Oxygen Meter.

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TABLE 1 - SOLUBILITY OF OXYGEN IN FRESH WATER

Temperature 'C	mg/l Dissolved Oxygen	Temperature 'C	mg/l Dissolved Oxygen
0	14.60	23	8.56
1	14.19	24	8.40
2	13.81	25	8.24
3	13.44	26	8.09
4	13.09	27	7.95
5	12.75	28	7.81
6	12.43	29	7.67
7	12.12	30	7.54
8	11.83	31	7.41
9	11.55	32	7.28
10	11.27	33	7.16
11	11.01	34	7.05
12	10.76	35	6.93
13	10.52	36	6.82
14	10.29	37	6.71
15	10.07	38	6.61
16	9.85	39	6.51
17	9.65	40	6.41
18	9.45	41	6.31
19	9.26	42	6.22
20	9.07	43	6.13
21	8.90	44	6.04
22	8.72	45	5.95

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

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TABLE 2 - ALTITUDE CORRECTION FACTOR

Atmospheric Pressure mmHg	or	Equivalent Altitude Ft.	Correction Factor
775		540	1.02
760		0	1.00
745		542	.98
730		1094	.96
714		1688	.94
699		2274	.92
684		2864	.90
669		3466	.88
654		4082	.86
638		4756	.84
623		5403	.82
608		6065	.80
593		6744	.78
578		7440	.76
562		8204	.74
547		8939	.72
532		9694	.70
517		10472	.68
502		11273	.66

Source: Derived from 15th Edition "Standard Materials for the Examination of Water and Wastewater."

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OPERATION PROCEDURE YSI MODEL 33  
S-C-T METER (SALINITY, CONDUCTIVITY, TEMPERATURE)

## 1.0 INTRODUCTION

The YSI Model 33 is a portable battery powered, transistorized instrument used to measure salinity, conductivity, and temperature in surface, ground water, and waste streams. Conductivity is expressed as micromhos/centimeter (umhos/cm; note: the "umhos/cm" on the meter is abbreviated "umho"). These are measurements of the electrical conductance the sample would show if measured between opposite faces of a 1 cm cube. Salinity is the number of grams of salt per kilogram of sample (0/00 = parts per thousand). This measurement assumes the sample contains a "standard" sea water salt mixture. The sample temperature is measured in degrees Celsius.

Salinity measurements are manually temperature compensated by direct dial. Conductivity measurements are not temperature compensated; however, a temperature function is provided on the instrument to aid with calculation of corrections. Also, when just temperature and conductivity are known, it is possible to calculate salinity, and when only temperature and salinity are known, it is possible to calculate conductivity.

## 2.0 Specifications

### 2.1 Conductivity

Ranges: 0-500, 0-5,000, and 0-50,000 umhos/cm with YSI 3300 Series Probes.  
Accuracy: + 2.5% max. error at 500, 5,000, and 50,000 plus probe. + 3.0% max. error at 250, 2,500 and 25,000 plus probe.  
Readability: 2.5 umhos/cm on 500 umho/cm range  
25 umhos/cm on 5,000 umho/cm range  
250 umhos/cm on 50,000 umho/cm range

Temperature Compensation: None

## 2.2 Salinity

Range: 0-40 0/00 in temperature range of -2 to +45°C  
Accuracy: Above 4°C, + 0.9 0/00 at 40 0/00 and  
+ 0.70/00 at 20 0/00 plus conductivity probe.  
Below 4°C, + 1.1 0/00 at 40 0/00 and  
+ 0.9 0/00 at 20 0/00 plus conductivity probe  
Readability: 0.2 0/00 on 0-40 0/00 range  
Temperature compensation: Manual by direct dial from -2 to +45°C.

## 2.3 Temperature

Range: -2 to 50°C  
Accuracy: + 0.1°C at -2°C; + 0.6°C at 45°C plus probe  
Readability: + 0.15°C at -2°C to + 0.37°C at 45°C

## 2.4 Power Supply

Two D-size alkaline batteries, Eveready E95 or equivalent, providing approximately 200 hours of operation.

## 2.5 Probe

YSI 3300 Series conductivity/temperature probe  
Normal Probe Constant:  $K = 5/\text{cm}$   
Accuracy: + 2% of reading for conductivity and salinity  
Error: + 0.1°C at 0°C and + 0.3° at 40°C

## 2.6 Instrument Ambient Range

Satisfactory operation -5 to +45°C. A maximum error of + 0.1% of the reading per °C change in instrument temperature can occur. The error is negligible if the instrument is readjusted to redline for each reading.

# 3.0 OPERATION PROCEDURE

## 3.1 Set Up

3.1.1 Adjust meter zero (if necessary) by turning the bakelite screw on the meter face so that the meter needle coincides with zero on the conductivity scale.

3.1.2 Calibrate the meter by turning the MODE control to REDLINE and adjusting the REDLINE control so the meter lines up with the redline on the meter face. If this cannot be accomplished, replace the batteries.

3.1.3 Plug the probe into the probe jack on the side of the instrument.

3.2 Temperature Measurements

3.2.1 Place probe in solution to be measured.

3.2.2 Set probe mode to Temperature.

3.2.3 Allow five minutes for probe to come to equilibrium with temperature of water before taking reading.

3.2.4 Read temperature on the bottom scale of the meter in degrees Celsius and record on appropriate form or log book.

3.3 Salinity Measurement

3.3.1 Transfer the temperature reading from step 2.2.4 to the °C scale on the instrument.

3.3.2 Switch the MODE control to the SALINITY position and read salinity on the red 0-40 0/00 meter range. Record on appropriate form or log book.

3.3.3 Depress the CELL TEST button. The meter reading should fall less than 2%; if greater, the probe is fouled and the measurement is in error. Clean the probe and re-measure.

3.4 Conductivity Measurement

3.4.1 Switch the MODE control to the X100 scale. If the reading is below 50 on the 0-500 range, switch to the X10 scale. If the reading is still below 50, switch to the X1 scale.

3.4.2 Read the meter scale and multiply the reading by appropriate scale factor. The answer is expressed in umhos/cm. Measurements are not temperature compensated. Record measurement on appropriate form or log book.

3.4.3 When measuring on the X10 and X100 scales, depress the CELL TEST button. The meter reading should fall less than 2%; if greater the probe is fouled and the measurement is in error. Clean probe and remeasure.

### 3.5 Error Calculation

#### 3.5.1 Temperature

Use Figure 1 to calculate error for probe and instrument versus °C meter reading.

Example: Meter reading 15°C  
Error from Figure 1 0.4°C  
Accuracy: 15°C  $\pm$  0.4°C for probe and instrument combined

#### 3.5.2 Conductivity

Use Figure 2 to determine the worst case conductivity error as a function of the conductivity reading for the probe and instrument combined.

Example: Meter Reading: 360 umhos/cm on X10 scale  
% Reading error from Figure 2  $\pm$  4.5%  
Accuracy: 3,600  $\pm$  162 umhos/cm for probe and instrument.

#### 3.5.3 Salinity

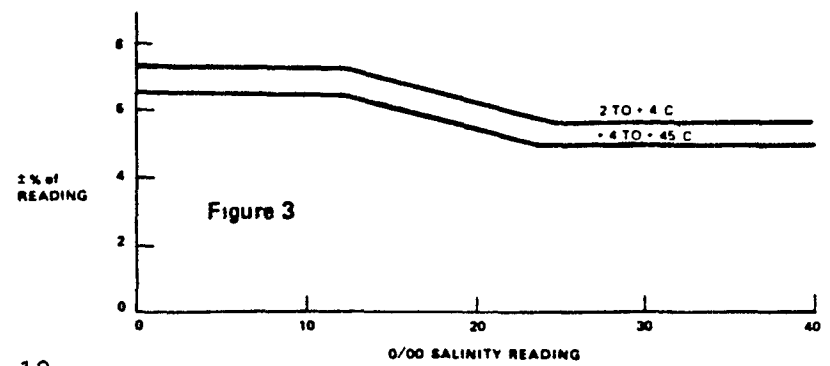
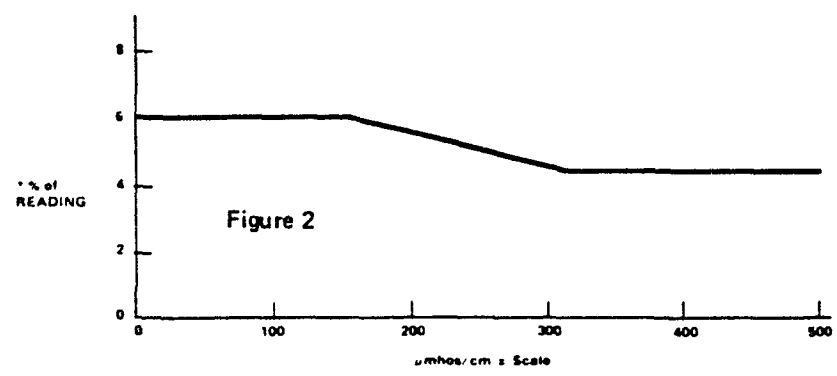
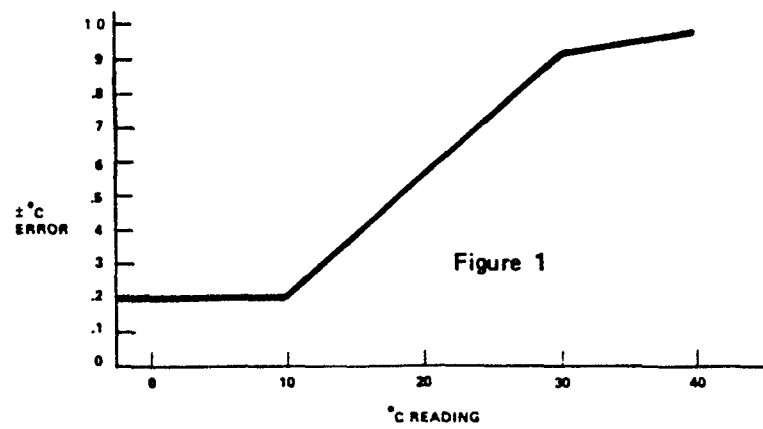
The salinity readings are a function of temperature and conductivity, therefore the accuracy is a function of both. Use Figure 3 to derive the total error of the temperature and conductivity probe.

Example: Meter Reading: 10 0/00, @ 10°C  
% of Reading Error  
From Figure 3: 6.5%  
Accuracy: 10 0/00  $\pm$  0.65 0/00 for all errors, combined worst case

### 3.0 REFERENCE

Yellow Springs Instrument Co., Inc. No date. Instructions for YSI Model 33 and 33M S-C-T meters.





## OPERATION PROCEDURE FOR HAAKEBUCHLER pH STICK

### 1.0 INTRODUCTION

The HaakeBuchler pH Stick is a portable pH monitoring instrument for determining pH in surface and ground waters, waste streams and other water quality applications. The pH Stick consists of a plastic bodied, gel filled, combination electrode and a miniaturized precision meter with liquid crystal display (LCD). The electrode incorporates a temperature sensor which provides automatic compensation for electrode temperature effects from 0 to 45°C.

### 2.0 SPECIFICATIONS

Instrument - None specified by the manufacturer.

Batteries - Uses seven V312HM mercury type (or equivalent) cells; 7.75 mm diameter; 3.50mm thickness; minimal voltage per cell of 1.35 volts.

### 3.0 OPERATION

#### 3.1 Field Calibration

The instrument requires field calibration prior to each use. Distilled water, buffer solution (pH 7) and pH 4 solution are required for the field calibration. All solutions must be at the same temperature. This reduces time to stabilize and improves accuracy.

To calibrate the instrument:

1. Remove the protective sheath and rinse the electrode in distilled water.
2. Place the electrode in the pH 7 buffer solution, depress the white operation button below the LCD display, and allow the reading to stabilize.
3. Adjust the pH 7 control using the tool on the end of the protective sheath. The pH 7 control is the upper most white control on the right side of the instrument. Adjust the pH control until the meter reads pH 7.
4. Rinse the electrode in distilled water.
5. Place the electrode in pH 4 solution, depress the white operation button, and allow the reading to stabilize.
6. Adjust the slope control (white control below pH 7 control on right side of instrument) until the meter reads the correct value of the pH 4 solution.
7. Rinse probe in distilled water.
8. Repeat steps 2 through 7.
9. Record calibration in field log or on appropriate form.

### 3.2 pH Measurements

To take pH measurements:

1. Remove protective sheath (if on) and rinse electrode in distilled water.
2. Place electrode in water sample, depress white control button, wait for reading to stabilize, and record reading in field log book or on appropriate form.
3. Rinse electrode in distilled water between each measurement and after the last sample is measured.

### 3.3 Instrument Storage

Always rinse the electrode in distilled water prior to replacing the protective sheath. Do not let the electrode dry out. The absorbent pad at the bottom of the sheath must be kept saturated with pH 7 buffer solution. Following each use, place buffer solution in the sheath prior to inserting the electrode. As a temporary measure, the instrument may be stored in distilled water. The distilled water should be replaced with pH 7 buffer at the earliest possible opportunity.

When not in use, place pH sticker in the wallet provided and store in dry place.

## PROCEDURE FOR DETERMINING TEMPERATURE OF GROUNDWATER

### 1.0 INTRODUCTION

The temperature of ground water is important for numerous applications. For example, temperature measurements are critical in identifying recharge from nearby surface-water sources. For accurate geochemical evaluation of equilibrium thermodynamics, it is desirable to know the temperature of the water in the aquifer within  $\pm 0.1^{\circ}\text{C}$ .

The temperature is recorded by a mercury-filled thermometer that is permitted to equilibrate in a sample that is continuously pumped into a dewar flask. The well should be pumped continuously until three identical consecutive readings of temperature are obtained.

The foregoing method may be used to measure the temperature of any ground water sample but is most accurate when the ambient temperature is within  $20^{\circ}\text{C}$  of the ground-water temperature. New or little-used wells may develop new producing zones during pumping, thereby varying the proportion of water entering the well from different depths and causing either an instantaneous change or continual drift in the temperature measurements. Drawdown may cause dewatering of certain beds and may cause a change in the temperature. Pumping time insufficient to allow equilibration of water temperature in the casing and pump column will also cause drifting of the temperature.

### 2.0 APPARATUS

1. Dewar flask (a thermos bottle with a narrow mouth is adequate).
2. Partial immersion thermometer calibrated in  $0.1^{\circ}\text{C}$  or  $0.2^{\circ}\text{F}$  with the usual range of  $0-50^{\circ}\text{C}$ , or any range expected in the ground water in the study area. Steel or brass armor should be used to minimize breaking the thermometer. The thermometer should be

accurate to +1°C as checked against the Bureau of Standards calibrated thermometer.

### 3.0 PROCEDURE

Samples should be collected as close to the wellhead as possible. A continuous stream of water should be conducted through a short plastic tube into the bottom of the flask, allowing a continuous flow of water from the flask. Readings should be taken after 5 minutes of flow at 1 minute intervals and recorded until no change is observed. If collection by continuous flow is impossible, the sample should be collected in the dewar flask and stoppered, with the thermometer inserted through the access hole. The first sample should be allowed to equilibrate 1 or 2 minutes and should then be discarded. The flask should be quickly refilled, and the temperature should be recorded immediately. This process should be continued until three identical consecutive readings are obtained. A box similar to that shown in Figure 1 is useful for carrying equipment and preventing breakage.

### 4.0 REFERENCE

USGS, 1984 National Handbook of Recommended Methods for Water-Data Acquisition.

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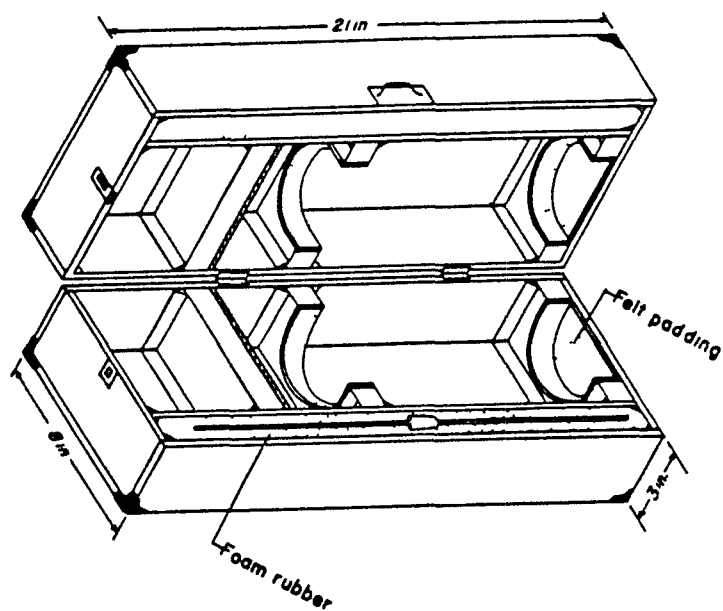


Figure 1 - Carrying box for thermometer, reading lens, and dewar flask

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## PROCEDURE FOR FILTRATION OF SAMPLES

### 1.0 INTRODUCTION

The sampler should carefully review any proposed procedures for filtering samples on site. Filtration of samples in which volatile organic constituents are of interest is not recommended, since filtration may strip these constituents from the sample. However, filtration of samples in which metals are the constituents of concern may be applicable depending on the proposed analytical method. If total recoverable methods are to be used, the sample should not be filtered. However, if measurement of dissolved metals is desired, the sample should be filtered on site.

The use of filtering in the dissolved method is designed to remove particulate matter drawn during sampling into the well, through the screen, from the surrounding geologic materials. These particulates may have adsorbed constituents that, once a preservative (particularly acid) is added, may become dissolved in the sample. Thus, if samples truly representative of in-situ ground-water quality are desired, filtering should be required. However, if the goal is simply to detect in the subsurface the presence of a constituent, filtering may not be recommended. Analyzing unfiltered samples may, accordingly, be particularly suitable for detection monitoring. However, establishment of a suitable background may become a problem because water-quality measurements may be strongly influenced by the design and construction of individual wells and the grain size distribution of the formation in which the intake of each well is located. The sampler will need to determine which method is most appropriate for each particular program. In some cases both filtered and unfiltered samples may be collected and compared.



If mineral precipitation is observed during filtration or if the chemical species of interest are suspected to be significantly present in colloidal form, an unfiltered acidified sample should also be collected and subsequently analyzed for the same parameters as the filtered sample. The containers for the filtered and unfiltered samples must be so labeled and appropriately identified in the field notes.

## 2.0 PROCEDURE

If filtration is required, the use of a 0.45 micron filter is generally considered appropriate. Occasionally well or surface waters may contain high concentrations of Total Suspended Solids (TSS) such that the 0.45 micron filters will clog during filtering. To avoid clogging, prefilters, available commercially, should be used in addition to the 0.45 micron filters. The filter should also be made of materials compatible with the chemical characteristics of the ground water samples.

Filtration of ground water samples will be performed when appropriate, as summarized in the table below.

<u>Analysis</u>	<u>Sample Collection</u>	
	<u>Filtered</u>	<u>Non-Filtered</u>
Volatile Organics	No	Yes
Total Metals or Ions	No	Yes
Dissolved Metals or Ions	Yes	Sometimes
	(acidify after filtration)	

## 3.0 REFERENCE

U.S. EPA, 1983. Test Methods for Evaluating Solid Waste. SW-846.

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U.S. EPA, 1983. Methods for the Chemical Analysis of Water and Wastes. March 1983. EPA-600/4-79-020.

Geotrans, Inc., 1983. RCRA Permit Writer's Manual: Ground Water Protection (40 CFR Part 264, Subpart F), EPA Contract no. 68-01-6464.

Scalf, M.R., McNabb, J.F., Dunlap, W.J., Cosby, R.L., Fryberger, J., 1981. Manual of Ground-Water Sampling Procedures. NWWA/EPA Series.

## PROCEDURES FOR WATER LEVEL MEASUREMENT

### 1.0 INTRODUCTION

Water-level measurements are fundamental to ground-water and solute transport studies. Some of the major uses of water-level data are to indicate the directions of ground-water flow and areas of recharge and discharge, to evaluate the effects of manmade and natural stresses on the ground-water system, to define the hydraulic characteristics of aquifers, and to evaluate stream aquifer relations. Uses for water level data are listed below:

1. Monitor the effect of mining or subsurface disposal operations.
2. Indicate the change in water level due to distribution or rate of regional groundwater withdrawal.
3. Show the relationship of groundwater to surface water.
4. Provide long-term records that can be used to evaluate the effect of management and conservation programs.
5. Estimate the amount, source, and area of recharge and estimate discharge.
6. Estimate the hydraulic characteristics of an aquifer.
7. Estimate evapotranspiration.
8. Estimate rate and direction of groundwater movement.
9. Delineate reaches of losing or gaining streams or canals.
10. Indicate the status or change in groundwater storage.

Water level should be measured at each site prior to well evacuation, sampling or other disturbance of the water table. Water level measuring techniques and their applications are summarized on Table 1.

[X indicates applicability of the methods]

Method	APPLICATION					Advantages	Disadvantages
	Water-level measurements 500 ft or less	Water-level measurements in 1,500-ft range	Observation of natural water-level fluctuation	Pumping test <sup>1</sup> where fluid-level changes are large	Hydraulic tests with small fluid-level changes (0.2 ft)		
Steel tape	X	X		possible	possible	Most precise method.	Slow, must remove from well to read; delicate.
Electric cable	X	X	Non-recording devices.	X	X	Rugged, simple, adequate field precision. <sup>2</sup>	Large instrumental error; required periodic calibration.
Air line	X	X		X	Lacks precision.	Fast, simple, not affected by foam. Precision depends on air gauge used.	Needs air compressor, air line must be airtight, limited by gauge inaccuracies.
Mechanical Recorder	X		X	X	X	Precise, permanent record.	Limited to relatively straight holes less than 1,000-ft deep.
Transducer Systems	X	X	X	X	Possible, lacks precision at present.	High-speed response, permanent record.	Needs frequent attention.

<sup>1</sup> Or other hydraulic test including packer test.

<sup>2</sup> High resolution (0.01 ft) when kept in hole for measuring small water level fluctuation.

Table 1 - Applications of Water Level Measuring Techniques  
(Modified from Garber and Koopman, 1968).

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## 2.0 MEASURING POINTS AND REFERENCE POINTS

For comparability, water-level measurements must be referenced to the same datum (elevation). The measuring point is measured in reference to land surface datum and is the most convenient place to measure the water level in a well. Measurement points change from time to time, especially on private wells. The measuring-point correction of a water-level measurement converts the measurement to a distance above or below land surface at the well.

The measuring point must be as permanent as possible, clearly defined, marked, and easily located. If at all possible, position the point so that a leveling rod can be set on it directly over the well and the measuring tape can hang freely when it is in contact with the measuring point. Frequently, the top of the casing is designated as the measuring point; because the top of the casing is seldom smooth and horizontal, a particular point should be designated and marked clearly with paint and, if permitted, the letters MP with an arrow.

The reference point for water-level measurements is an arbitrary datum established by permanent marks set on or near the well. It is used to check the measuring point, and its greatest value is in re-establishing a measuring point if one is destroyed or changed.

## 3.0 PROCEDURES

### 3.1 Graduated Steel Tape

The graduated steel tape method is considered to be the most accurate method for measuring the water level in nonflowing wells. Steel surveying tapes in lengths of 100, 200, 300, 500, and 1,000 feet are commonly used. A black tape is better than a chromium-plated tape. The tapes are mounted on hand-cranked

reels up to 500-ft lengths; for greater depths a motor-driven tape drive is usually required. A slender weight is attached to the ring at the end of the tape to insure plumbness and to permit some feel for obstructions.

The lower few feet of tape is chalked by pulling the tape across a piece of blue carpenter's chalk. The wet chalk mark identifies the portion of the tape that was submerged. Lower the graduated steel-tape from the measuring point at the top of the well until a short length of the tape is submerged. The weight and tape should be lowered into the water slowly to prevent splashing. Submergence of the weight and tape may temporarily cause a water-level rise in wells or piezometers having very small diameters. This effect can be significant if the well is in materials of very low hydraulic conductivity. Under dry surface conditions, it may be desirable to pull the tape from the well by hand, being careful not to allow it to become kinked, and reading the water mark before rewinding the tape onto the reel. In this way, the water mark on the chalked part of the tape is rapidly brought to the surface before the wetted part of the tape dries. In cold regions, rapid withdrawal of the tape from the well is necessary before the wet part freezes and becomes difficult to read. Read the tape at the measuring point, and then read the water mark on the tape. The difference between these two readings is the depth to water below the measuring point.

Errors resulting from the effects of thermal expansion of tapes and of stretch due to the suspended weight of the tape and plumb weight can become significant at high temperatures and for measured depths in excess of 1,000 feet.

As a standard of good practice, the observer should make two measurements. If two measurements of static water level made

within a few minutes do not agree within about 0.01 or 0.02 foot (generally regarded as the practical limit of precision) in observation wells having a depth to water of less than a couple hundred feet, continue to measure until the reason for the lack of agreement is determined or until the results are shown to be reliable. Where water is dripping into the hole or covering its wall, it may be impossible to get a good water mark on the chalked tape.

### 3.2 Electrical Methods

Many types of electrical instruments have been devised for measuring water levels; most operate on the principle that a circuit is completed when two electrodes are immersed in water. Some instruments consist of a single conductor that is lowered into the well where the metal well casing is used as the second conductor. More commonly, a two-conductor cable and special probe are used. Various forms of electrolytic cells using two electrodes of dissimilar metals have been used, but current is more commonly supplied by batteries.

Ordinarily, two-conductor electric tapes are 500-ft long and are mounted on a hand-cranked reel that contains space for the batteries and some device for signaling when the circuit is closed. Electrodes are generally contained in a weighted probe that keeps the tape taut while providing some shielding of the electrodes against false indications as the probe is being lowered into the hole. The electric tapes generally are marked at 5-ft intervals with clamped-on metal bands.

Before lowering the probe in the well, the circuitry can be checked by dipping the probe in water and observing the indicator. The probe should be lowered slowly into the well

until contact with the water surface is indicated. The electric tape is marked at the measuring point and partly withdrawn; the distance from the mark to the nearest tape band is measured and added to (or subtracted from) the band reading to obtain the depth to water. It is good practice to take a second or third check reading before withdrawing the electric tape from the well. The tape should not rub across the top of the casing because the metal bands can become displaced.

Electric tapes are more cumbersome and inconvenient to use than the wetted-tape method, and they normally give less accurate results. In some situations, however, they are superior. Where water is dripping into the hole or covering its walls, it may be impossible to get a good water mark on the chalked tape. In wells that are being pumped, particularly with large-discharge pumps, the splashing of the water surface makes consistent results by the wetted-tape method impossible. Where a series of measurements are needed in quick succession, such as in pumping tests, electric tapes have the advantage of not having to be removed from the well for each reading. Electric tapes are also safer to use in pumping wells because the water is sensed as soon as the probe reaches the water surface and there is less danger of lowering the tape into the pump impellers.

Independent electric tape measurements of static water levels using the same tape should agree within  $\pm 0.04$  foot for depths of less than about 200 feet. At greater depths, independent measurements may not be this close. For a depth of about 500 feet, the maximum difference of independent measurements using the same tape should be within  $\pm 0.1$  foot.

In wells having a layer of oil floating on the water, the electric tape will not respond to the oil surface and, thus, the



fluid level determined will be different than would be determined by a steel tape. The difference depends on how much oil is floating on the water. A miniature float-driven switch can be put on a two-conductor electric tape that permits detection of the surface of the uppermost fluid. The electric tape should be decontaminated after measurements in each well.

It is especially important to check the electric line length by measuring with a steel tape after the line has been used for a long time or after it has been pulled hard in attempting to free the line. Some electric lines, especially the single line wire, are subject to considerable permanent stretch. In addition, because the probe is larger in diameter than the wire, the probe can become lodged in a well. Some operators attach the probe by twisting the wires together by hand and using only enough electrical tape to support the weight of the probe. In this manner, the point of probe attachment is the weakest point of the entire line. Should the probe become "hung in the hole", the line may be pulled and breakage will occur at the probe attachment point, allowing the line to be withdrawn.

### 3.3 Air Line

The air line method is especially useful in pumped wells where water turbulence may preclude using more precise methods. A small diameter air-type tube of known length is installed from the surface to a depth below the lowest water level expected. Compressed air (compressor, bottled air, or tire pump) is used to purge the water from the tube. The pressure, in pounds per square inch (psi), needed to purge the water from the air line multiplied by 2.31 (feet of water for one psi) equals the length in feet of submerged air line. The depth to water below the center of the pressure gage can be easily calculated by

subtracting the length of air line below the water surface from the total length of air line (assuming the air line is essentially straight).

Accuracy depends on the precision to which the pressure can be read. The accuracy of an air line or pressure gage measurement depends primarily on the accuracy and condition of the gage but is normally within one foot of the true level as determined by means of a steel-tape measurement. The air lines themselves, however, have been known to become clogged with mineral deposits or bacterial growth or to develop leaks and consequently yield false information. A series of air-line measurements should be checked periodically by the use of a steel tape or an electric water-level indicator.

The air line and any connections to it must be airtight throughout its entire length. If the line is broken or leaky, large errors may occur. A long-term increase in air line pressure may indicate a gradual clogging of the air line. A relatively sudden decrease in air line pressure may indicate a leak or break in the air line. Air line pressures that never go above a constant low value may indicate that the water level has dropped below the outlet orifice of the air line. To minimize the effect of turbulence, the lower end of the air line should be at least five feet above or below the pump intake. Corrections should be made for fluid temperatures much different from 20°C and for vertical differences in air density in the well column for cases where the depth to water is very great.

### 3.4 Recording Devices

Devices for recording changes in water levels may be mechanical, electronic, or electromechanical. A further distinction is the

manner in which the device detects changes. A float or an electromechanically actuated water-seeking probe may be used to detect vertical changes of the water surface in the hole, or a mechanical or electrical pressure gage submerged several feet below the water surface may be used to detect changes in fluid pressure resulting from water-level changes. The principal advantage of pressure-sensing devices is that they may be used in packed or otherwise sealed-off zones in a well. In addition, their response to rapid changes of fluid pressure is generally better than that of mechanical devices. An increasing number of digital punched-tape recorders that record levels at prescribed times are being used on observation wells. However, the simplest recording device commonly in use consists of a recording chart drum rotated mechanically by a free float that follows the water level, while a clock drive moves a recording pen horizontally across the chart.

Where depth to water is more than a few feet below the top of the casing, special care must be taken to minimize friction between the float cable and the walls of the well. The float selected should be the largest diameter that can be accommodated by the casing. The error resulting from float-line drag along the well casing is larger than the error caused by float drag. If the clearance between the float and the casing is small, the float cable should be arranged so that the counterweight does not have to pass the float, but is always above or below the water level. When the counterweight is immersed, a little extra weight should be used to counteract the water's buoyancy.

Water-level recorders should be protected from the weather and vandalism by a suitable shelter that is solidly anchored. The part of a recorder most susceptible to malfunction is the clock. To insure continuous records, field personnel who visit recorders

should carry spare clocks. Malfunctioning clocks should be sent to a reliable clock-repair service for maintenance, and all clocks should be cleaned and adjusted periodically.

During each visit to a recorder installation, the water level should be measured (preferably by the wetted-tape method) and the measurement compared with the recorded value and the appropriate adjustments made.

In flowing wells where the static water level is above the top of the casing, float-activated recorders cannot be used. If the well can be shut in, however, a pressure recorder can be used.

### 3.5 Special Procedures for Immiscible Fluids

At those facilities where monitoring to determine the presence or extent of immiscible contaminants is required, the sampler will need to use special procedures for the measurement of fluid levels. The procedures required will depend on whether light immiscibles that form lenses floating on top of the water table or dense immiscibles that sink through the aquifer and form lenses over lower permeability layers are present.

In the case of light immiscibles, measurements of immiscible fluid and water level usually cannot normally be accomplished by using normal techniques. For example, a chalked steel tape measurement will only indicate the depth to the immiscible fluid (not the depth to water) and a conventional electric tape will not generally respond to nonconducting immiscible fluids. Similar problems are found with other techniques.

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To circumvent these problems, the use of special techniques and equipment can be specified. These techniques have been specially developed to measure fluid levels in wells containing immiscible fluids, particularly petroleum products. One method is similar to the chalked steel tape method. The difference is the use of a special paste or gel rather than ordinary carpenters chalk. Such indicator pastes, when applied to the end of the steel tape and submerged in the well, will show the top of the oil as a wet line and the top of the water as a distinct color change. Another method, similar to the electric tape method, uses a dual purpose probe and indicator system. The probe can detect the presence of any fluid (through the wetting effect) and can also detect fluids that conduct electricity. Thus, if a well is contaminated with low density, nonconducting immiscible fluids such as gasoline, the probe will first detect the surface of the gasoline, but it will not register electrical conduction. However, when the probe is lowered deeper to contact water, electrical conduction will be detected.

Both of these methods have disadvantages. The paste is not very effective with heavier and less refined petroleum products. Also, it can be a cumbersome technique when many wells must be measured owing to tape decontamination between wells. Also, this method cannot be used when sampling for constituents normally found in the paste.

It is important to note that water levels obtained in this situation are not suitable without further interpretation for determining hydraulic gradients. To use those data for determining hydraulic gradients, the differences in density between the light immiscible and water have to be accounted for.

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Measuring fluid levels in wells screened in lenses of dense immiscibles resting on a low permeability formation is somewhat easier, provided the immiscible fluid is nonconducting. The top of the dense layer can be identified by simply using an electric sounder. As an electric sounder passes from groundwater into the immiscible phase, the detection unit will deactivate because the fluid will no longer conduct electricity. Special pastes or gels may also be available for this purpose. An additional method for determining the interface of a denser-than-water fluid may be appropriate. An hydrometer having a density greater than water but less than the immiscible fluid may be used. Although it is a delicate procedure, the hydrometer should sink through the water but float on the immiscible fluid. Water levels in such wells can be measured by using normal procedures.

#### 4.0 REFERENCES

- USGS. 1984. National Handbook of Recommended Methods for Water-Data Acquisition.
- USGS. 1983. RCRA Permit Writer's Manual: Ground Water Protection (40 CFR Part 264 Subpart F). Geotrans Inc., prepared EPA.

## PROCEDURE FOR WELL EVACUATION

### 1.0 INTRODUCTION

To obtain a representative sample of the groundwater it must be understood that the composition of the water within the well casing and in close proximity to the well is probably not representative of the overall groundwater quality at that sampling site. This is due to the possible presence of drilling contaminants near the well and because important environmental conditions such as the oxidation-reduction potential may differ drastically near the well from the conditions in the surrounding water-bearing materials. For these reasons it is highly desirable that a well be pumped or bailed until the well is thoroughly flushed of standing water and contains fresh water from the aquifer. The recommended amount of purging before sampling is dependent on many factors including the characteristics of the well, the hydrogeological nature of the aquifer, the type of sampling equipment being used, and the parameters being sampled.

A common procedure is to pump or bail the well until a minimum of 2 to 10 bore-volumes have been removed. Two alternative approaches are: (1) monitor the water level in the well while pumping. When the water level has "stabilized" most if not all of the water being pumped is coming from the aquifer; (2) monitor the temperature, conductivity, or pH of the water while pumping. When these parameters "stabilize" it is probable that little or not water from casing storage is being pumped.

The use of an indicating analysis such as pH, temperature, redox potential, or, most commonly, conductivity, may be the most accurate and reliable method of assuring complete well purging. The technique

is easily implemented in the field and gives a rapid and positive indication of changes in the well bore water. This change in the water character and subsequent stabilization can normally be interpreted as evidence that sufficient purging has occurred. It should be noted that the sensitivity of these parameters to changes as a result of exposure of groundwater to surface level conditions (i.e., changes in the partial pressure of dissolved gases or the conditions of the purging system) make in-situ monitoring desirable. An alternative to this would be to conduct these measurements in a closed cell attached to the discharge side of the pump system.

Other factors which will influence the amount of purging required before sampling include the pumping rate and the placement of the pumping equipment within the column of water in the well bore. For example, recent studies have shown that if a pump is lowered immediately to the bottom of a well before pumping, it may take some time for the column of water above it to be exchanged if the transmissivity of the aquifer is high and the well screen is at the bottom of the casing. In such cases the pump will be drawing water primarily from the aquifer.

Purging from just below the water surface insures a more complete removal of the casing water than by withdrawal from well below the surface. When purging does occur from just below the surface, satisfactory results can be obtained at any of a wide range of pumping rates with either a peristaltic or a submersible pump.

Because of the potential for further environmental contamination, planning for purge water disposal is a necessary part of well monitoring. Alternatives range from dumping it on the ground (not back down the well) to full containment, treatment, and disposal. If the well is believed to be contaminated, the best practice is to contain the purge water and store it until the water samples have been



analyzed. Once the contaminants are identified, appropriate treatment requirements can be determined.

## 2.0 METHODS

There are many methods available for well purging. In some cases bailing will suffice; however, it can become tedious and labor intensive in deep or large diameter wells. Submersible pumps are often the best choice, but most that are readily available to investigators are heavy, awkward and will not fit smaller diameter wells. Models have been on the market for the past few years that will fit inside a 2-inch diameter well; however, they can be costly and not always easy to come by. Gas pressure lift systems are useful in many instances. They are generally light, easy to install, and are useful in many instances. They can be powered by several different pressure systems, usually compressed nitrogen or air. The effect of the contact between the pressure gas and the groundwater usually results in changes in the dissolved gas content. As a result pH, conductivity, or other analysis used to determine purge completion must be conducted down hole. Recent developments in the use of pumps powered by compressed gas have shown promise. Although these too have large gas volume demands when operated at substantial depths, some versions, such as the one built and tested by the NCASI, can also be used for sample collection. Peristaltic pumps are widely used for purging of wells with water levels close to the surface (less than 8 meters). They are reasonably portable, light, and easily adaptable to ground level monitoring of purge indicator parameters by attaching a flow-through cell. These pumps require a minimum of down hole equipment and can easily be cleaned in the field; or the entire tubing assembly can be changed for each well.

### 3.0 PROCEDURES

#### 3.1 Purging With a Peristaltic Pump

The peristaltic pump can be used for the presample purging of groundwater monitor wells. The use of a peristaltic pump for well purging is particularly advantageous since the same system can later be utilized for sample collection. The application, however, is limited to wells with the capability for peristaltic action.

Procedures for use:

1. Using clean equipment, sound well for total depth and water level, then calculate the fluid volume in the casing ("casing volume").
2. Determine depth from casing top to mid-point of screen, or from casing top to the well section open to the aquifer. (Consult drillers log or sound for bottom.)
3. If depth to mid-point of screen is in excess of 8 meters, choose alternate system.
4. Lower intake into the well to a short distance below the water level and begin water removal. Collect or dispose of purged water in an acceptable manner. Lower suction intake, as required, to maintain submergence.
5. Measure rate of discharge frequently. A bucket and stopwatch are most commonly used.
6. Purge a minimum of two casing volumes or until discharge, pH, temperature, or conductivity stabilize.
7. After pumping, monitor water level recovery. Recovery rate may be useful in determining sample rate.

### 3.2 Purging With a Gas Pressure Displacement System

A pressure displacement system consists of a chamber equipped with a gas inlet line, a water discharge line and two check valves (see Figure 1). When the chamber is lowered into the casing, water floods it from the bottom through the check valve. Once full, a gas (i.e., nitrogen or air) is forced into the top of the chamber in sufficient amounts to result in the upward displacement of the water out the discharge tube. The check valve in the bottom prevents water from being forced back into the casing and the upper check valve prevents water from flowing back into the chamber when the gas pressure is released. This cycle can be repeated as necessary until purging is complete. The pressure lift system is particularly useful when the well depth is beyond the capability of a peristaltic pump. The water is displaced up the discharge tube by the increased gas pressure above the water level. The potential for increased gas diffusion into the water makes this system unsuitable for sampling for volatile organic or most pH critical parameters.

#### Procedures for use:

1. Using clean noncontaminating equipment (i.e., an electronic level indicator) determine the water level in the well then calculate the fluid volume in the casing.
2. Determine depth to midpoint of screen or depth to well section open to the aquifer (consult drillers log).
3. Lower displacement chamber until top is just below water level.
4. Attach gas supply line to pressure adjustment valve on cap.
5. Gradually increase gas pressure to maintain discharge flow rate.
6. Measure rate of discharge frequently. A bucket and stopwatch are usually sufficient.

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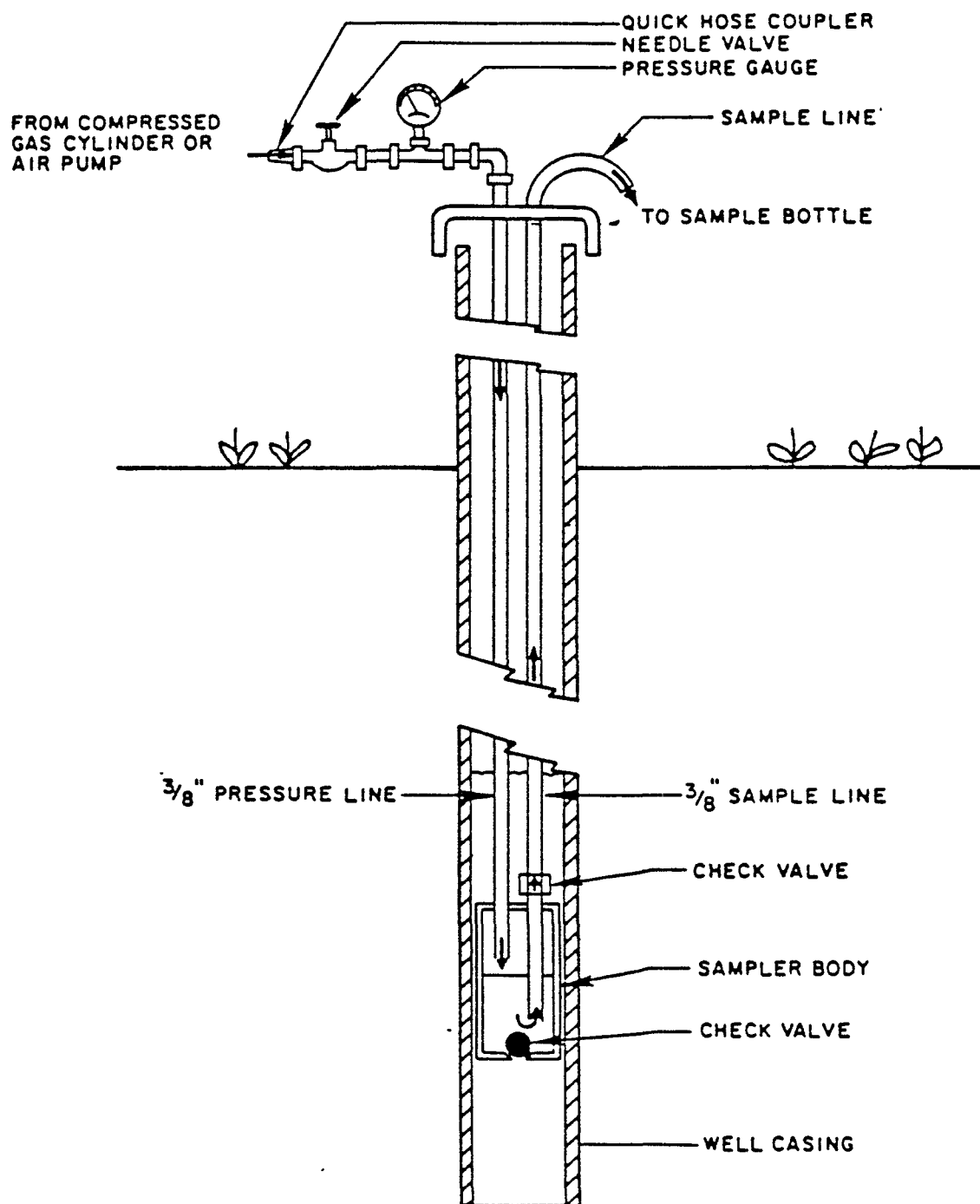


Figure 1 Gas pressure displacement system.

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7. Purge a minimum of two casing volumes or until discharge characteristics stabilize (see discussion on well purging).
8. After pumping, monitor water level recovery. Recovery rate may be useful in determining sample rate.

#### 4.0 REFERENCES

- U.S. Environmental Protection Agency, "Procedures Manual for Ground Water Monitoring at Solid Waste Disposal Facilities," EPA-530/SW-611, August 1977.
- U.S. Environmental Protection Agency, "Sampling at Hazardous Materials Incidents", EPA Hazardous Response Support Division, Cincinnati, 1984.

## CONSIDERATIONS FOR SAMPLE WITHDRAWAL FROM WELLS

### 1.0 INTRODUCTION

Once the well has been sufficiently purged (procedure 5619008) the actual sampling should begin as soon as the water level begins to approach its pre-purged level. Sampling for volatile organics may begin even sooner, before substantial volatilization begins. If recovery is very slow, it may be necessary to wait several hours or even until the following day before sufficient volume is available for all the necessary analyses. In this instance a volatile organics sample set may be collected soon after completion of the purging process and a second set collected with the remaining samples. When a pump is used for sample collection, its rate should be controlled to closely match the transmissivity of the formation. Excessive draw down of the well during sampling may result in nonrepresentative samples due to changes in groundwater flow.

The major consideration for sample withdrawal is ensuring that the sample is not altered or contaminated during withdrawal from the well. The sampling equipment must use materials compatible with the goals of the particular monitoring program. These materials must neither leach nor adsorb constituents of interest at levels that might be considered significant. Similarly, sampling equipment must be dedicated to individual wells or be capable of being fully disassembled and fully cleaned between sampling events.

Care should be taken to ensure that sampling equipment is lowered into the well with a proper cord. Sampling equipment should be lowered into the well by using single strand (non-plaited) wire of a material that does not conflict with the constituents of interest. Stranded or plaited wire is extremely difficult to decontaminate as pollutants may remain between strands after cleaning and could cause cross

contamination. Plastic-coated wire is often used, except where certain organics are being considered. Teflon-coated wire is the best, although most expensive, choice.

It is also important not to allow the use of methods that allow excessive exposure to the atmosphere or other gases when they might influence the measurement of specific constituents. Exposure of the sample to the atmosphere or other gases may result in changes in the concentration of dissolved gases in the sample. These changes can alter the pH of the sample, which in turn may precipitate the dissolution of specific constituents, particularly metals, and influence water quality measurements. Similarly, changes in the redox potential and, consequently, in the chemical species present may result. Exposure of the sample to the atmosphere or reduced pressures may also result in the degassing and loss of volatile organics from the sample.

When specifying methods for removal of water from monitoring wells, the sampler should examine the proposed methods to ensure that they are suitable both for sample collection and for well evacuation prior to sampling. In general, evacuation methods require greater pumping rates while sample collection methods emphasize to a greater extent the preservation of sample integrity. In many instances, one method will be suitable for both purposes. However, in some cases the use of different methods for well evacuation and sample collection may be appropriate. When examining methods used solely for well evacuation, the sampler should not allow the use of any method that might contaminate or otherwise alter the quality of water remaining in the well. Such an alteration would prevent the subsequent removal of samples representative of ground-water quality.

## 2.0 SAMPLING STEPS

When sampling a monitoring well, the following procedure should be followed:

1. Check the well for above ground damage.
2. Remove the well cap (a wrench may be needed).
3. Measure and record the depth to water and the time of measurement.
4. Measure the total depth of the well.
5. Remeasure and record the depth of water after a lapse of 4 to 8 minutes following initial measurement and record the depth to water and time of measurement.
6. If successive measurements show essentially no difference, continue the sampling procedure. Where the level change is greater than 1/100th ft, delay the remaining procedures until the change observed and recorded is less than that figure.
7. Determine the amount of water in the well (depth of water x cross sectional area).
8. Clean the sampler as described in the following Section (3.0) immediately prior to inserting it into a well.
9. Collect samples using a bailer, pump, or positive pressure sampler dependent upon the general nature of the contaminants of concern. Sampler materials of construction shall be specifically selected for each contaminant.

## 3.0 SAMPLER CLEANING

Sampler cleaning is to be performed immediately prior to sampling from any well. Any portion of the sampling device which contacts contaminated water shall be cleaned or disposed of between wells. For example, the cable used for bailing shall be subjected to the same cleaning requirement for a length equal to at least twice the depth to



the water surface. Where pumps are used, short sections of sample tubing may be disposed of rather than cleaned. The following procedure is followed for cleaning samplers and equipment:

- o Rinse with clean tap water.
- o Rinse with reagent grade methanol and allow to air dry.
- o Rinse at least once with clean tap water.

If appropriate the above procedure can be preceded with a wash with soap (Alconox or Liqinox). Line and cables used to lower samplers into monitoring wells will be disposed of in an appropriate manner dictated by the On-site Coordinator or cleaned and rinsed using the procedures outlined above for cleaning sampling devices. Persons obtaining groundwater samples will wear chemically inert protective gloves to prevent skin contact with sampling devices, lines and cables and potentially contaminated groundwater.

#### 4.0 IMMISCIBLE FLUIDS SAMPLE WITHDRAWAL

Recovery of ground water and immiscible fluid samples requires special procedures. Unlike in normal wells, evacuation immediately prior to sampling is not appropriate. Evacuation will normally result in mixing of the two phases, and several days may be required before the two fluids separate and water and immiscible fluid levels stabilize. If evacuation is desired, a few days or possibly a week or more, may be needed for the well to stabilize before sampling.

A sample of the floating immiscible may then be taken by using a bailer that fills from the bottom. Care should be taken to lower the bailer just through the petrochemical layer but not significantly down into the underlying ground water. Samples of the ground water at the bottom of the screen and at some intermediate location, such as the mid-point of the screen, may also be obtained with a bailer. However, in order to avoid mixing the waters, a separate casing can

be temporarily lowered inside the permanent well casing. This casing can be equipped with an easily removed cap on the bottom so that no fluid enters the casing until it has reached the desired depth for sampling. The cap is then knocked free of the bottom of the casing, allowing water entering from that specific depth to be sampled by bailer. At significant depths below the petrochemicals, several full bailers of water may be withdrawn and discarded before the sample is taken to obtain a fresh formation sample. However, it may be impractical to continually knock free caps from the bottom of the insert casing. In some cases, the existing well may no longer be screened in portions of the aquifer containing only water. Consequently, in cases where a large layer of immiscibles develops on top of the water table, it may be best to install a second well screened only in that portion of formation below the lens of floating immiscibles.

Sampling of dense immiscible may be accomplished by using a grab sampler. Pumps with intakes located in immiscible fluid may also be used. If a pump is used, it must be operated at a sufficiently low rate to ensure that no mixing occurs.

## 5.0 PROCEDURES

A variety of methods are available for sampling and well evacuation, including bailers, suction lift pumps such as the centrifugal and peristaltic pumps, and gas lift methods. Positive displacement or submersible pumps such as centrifugal, piston, gas squeeze, or jet pumps are also available. These methods use differing principles for operation and have distinct and frequently different advantages and disadvantages when applied to sampling and well evacuation. Each is discussed individually below and a summary of their advantages and disadvantages is provided in Table 1. Frequently, several methods will be available that can provide a suitable means for well

TABLE 1 Advantages and Disadvantages of Monitoring Well Sampling Equipment (adopted from Larson (1981a))

Type	Advantages	Disadvantages
A. Baller	Can be constructed in a wide variety of diameters	Time consuming sampling; sometimes impractical to properly evacuate casing before taking actual samples
	Can be constructed from a wide variety of materials	Transfer of water to sample bottle may result in aeration
	No external power source required	
	Extremely portable	
	Low surface area to volume ratio, resulting in a very small amount of outgassing of volatile organics while sample is contained in baller	
	Easy to clean	
	Readily available	
B. Suction lift Pump (Centrifuged, Peristaltic)	Inexpensive	
	Relatively portable	Sampling is limited to situations where water levels are within about 20 ft. from ground surface
	Readily available	
	Inexpensive	Vacuum effect can cause the water to lose some dissolved gas and volatile organics
		In some cases not constructed with materials compatible with sampling certain constituents

Table 1 (Continued)

Type	Advantages	Disadvantages
C. Gas lift samplers	<p>Relatively portable</p> <p>Readily available</p> <p>Inexpensive</p> <p>Very suitable for well development</p>	<p>Generally not considered appropriate method for acquisition of water samples for detailed chemical studies owing to degassing</p> <p>Regardless of the gas utilized, changes in CO<sub>2</sub> concentrations make this method unsuitable for sampling for pH sensitive parameters</p> <p>Aeration of water remaining in well frequently makes method unsuitable for well evacuation</p> <p>If air is used, oxygenation is impossible to avoid unless elaborate precautions are taken (only a very small amount of oxygen is required to cause a water sample to attain saturation with respect to oxygen)</p>
D. Submersible pumps	<p>Wide range in diameters</p> <p>Various materials are available</p> <p>Fairly portable</p> <p>Depending upon size of pump and pumping depths, relatively large pumping rates are possible</p> <p>Positive displacement minimizes loss of volatiles during pumping</p> <p>Readily available</p>	<p>Conventional units are unable to pump sediment-laden water without incurring damage to the pump</p> <p>Relatively expensive</p>

Table 1 (Continued)

Type	Advantages	Disadvantages
E. Gas-Operated squeeze pump (Middelburg type)	<p>Can be constructed in diameters as small as one inch</p> <p>Can be constructed from a wide variety of materials</p> <p>Relatively portable</p> <p>Fair range in pumping rates is possible</p> <p>Driving gas does not contact water sample, eliminating possible contamination or gas stripping</p> <p>Positive displacement minimizes loss of volatiles</p>	<p>Gas source required</p> <p>Large gas volumes and long cycles are necessary for deep operation</p> <p>Pumping rates are not as great as with suction or jet pumps</p> <p>Commercial units are relatively expensive, - pumps cost around \$300.00, while central gas unit cost around \$15.0</p>
F. Gas driven piston pump	<p>Isolates the sample from the operating gas</p> <p>Requires no electrical power source</p> <p>Operates continuously and reliably over extended periods of time</p> <p>Uses compressed gas economically</p> <p>Can be operated at pumping heads in excess of 500 m</p> <p>Positive displacement minimizes loss of volatiles</p>	<p>Relatively expensive; in excess of \$3,000 for the continuously operating unit</p> <p>Particulate material may damage or inactivate pump unless the suction line is filtered</p> <p>Low pumping rates</p>
G. Jet pumps	<p>Capable of producing high yields</p> <p>Commonly used in shallow domestic wells</p> <p>Available for relatively small diameter wells</p>	<p>Requires priming and mixing sample with water circulating in pump, greatly limiting any use in monitor well applications</p> <p>Use of venturi may result in stripping of volatiles</p>

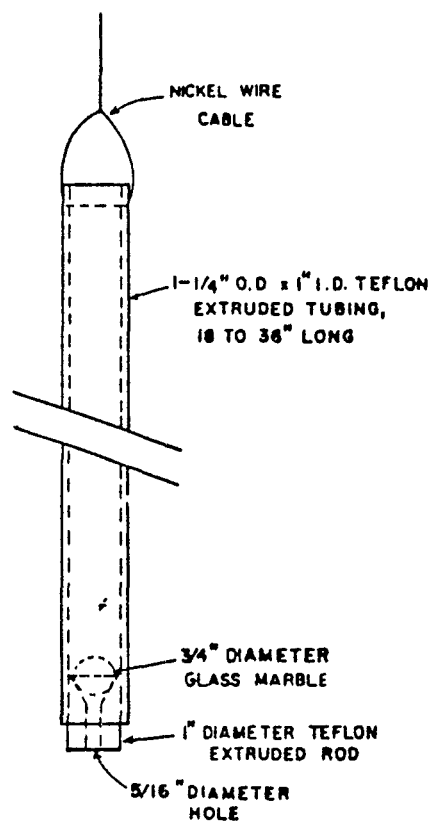


Figure 1 - Teflon Bailer

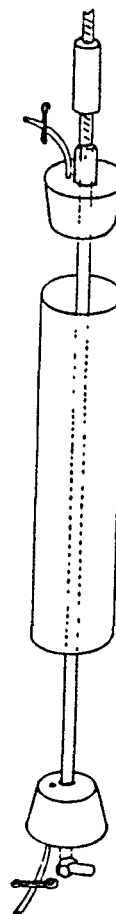


Figure 2 - Modified Kemmerer Sampler

evacuation and sample collection in a particular monitoring program. In such cases, the sampler will want to base the choice of method partly on such factors as monitoring well diameter, water level, pumping volume and rate requirements, accessibility of the site, ease of use, and cost. All of these factors are legitimate and should be considered as long as they do not unduly compromise the goals of the sampling program.

### 5.1 Bailers

The bailer is one of the oldest and simplest methods available. Bailers consist of a container attached to a cable that is lowered into the well to retrieve a sample. They can be of various designs. The simplest is a weighted bottle or basally capped length of pipe that fills from the top as it is lowered into the well. More sophisticated bailers have a check valve located at the base that allows water to enter from the bottom as it is lowered into the well (see Figure 1). When the bailer is lifted, the check valve closes, allowing water in the bailer to be brought to the surface. More sophisticated bailers are available that remain open at both ends while lowered into the well but can be sealed at both top and bottom by activating a triggering mechanism from the surface. This allows more reliable sampling at discrete depths within a well. Perhaps the best known bailer of this design is the Kemmerer sampler, shown in Figure 2.

Bailers generally provide an excellent means for collecting samples from monitoring wells. They can be constructed from a wide variety of materials compatible with the parameter of interest. Since they are relatively inexpensive, bailers can be easily dedicated to an individual well to minimize cross contamination during sampling. If not dedicated to a well, they can be easily cleaned to prevent cross contamination. Unfortu-

nately, bailers are frequently not suited for well evacuation because of their small volume.

This device is particularly useful when samples must be recovered from depths greater than the range (or capability) of suction lift pumps, when volatile stripping is of concern, or when well casing diameters are too narrow to accept submersible pumps. It is the method of choice for the collection of samples which are susceptible to volatile component stripping or degradation due to the aeration associated with most other recovery systems. Samples can be recovered with a minimum of aeration if care is taken to gradually lower the bailer until it contacts the water surface and is then allowed to sink as it fills. The primary disadvantages of bailers are their limited sample volume and inability to collect discrete samples from a depth below the water surface.

The following general procedures should be adhered to for sample withdrawal using a bailer.

1. Lower bailer slowly until it contacts the water surface, then allow it to sink and fill with a minimum of surface disturbance.
2. The cable supporting the sampler should be prevented from coming in contact with the water in the well. The bailer should not come in contact with any materials outside of the well casing. Do not allow the bailer line to contact the ground.

## 5.2 Suction Pumps

As their name implies, suction pumps operate by creating a partial vacuum in a sampling tube. This allows the pressure exerted by the atmosphere on the water in the well to force water up the tube to the surface. Accordingly, these pumps are located at the surface and require only that a transmission tube be



lowered into the well. Unfortunately, their use is limited by their reliance on suction to depths of 20 to 25 ft, depending on the pump. In addition, their use may result in out-gasing of dissolved gases or volatile organics and is therefore limited in many sampling applications. In spite of this, suction methods may provide a suitable means for well evacuation since the water remaining in the well is left reasonably undisturbed.

A variety of pumps are available that operate on this principle, but the most commonly suggested pumps for monitoring purposes are the centrifugal and peristaltic pumps. In the centrifugal pump, the fluid is displaced by the action of an impeller rotating inside the pump chamber. This discharges water by centrifugal force. The resulting pressure drop in the chamber creates a suction and causes water to enter the intake pipe in the well. These pumps can provide substantial yields and are readily available and inexpensive. The disadvantages are that they require an external power source and may be difficult to clean between sampling events. In addition, the materials with which these pumps are constructed may frequently be incompatible with sampling certain constituents. However, their substantial pumping rates make them suitable for well evacuation.

Peristaltic pumps operate in a similar manner to centrifugal pumps but displace the fluid by mechanical peristalsis. A flexible transmission line is mounted around the perimeter of the pump chamber and rotating rollers compress the tubing, forcing fluid movement ahead (the peristaltic effect) and inducing suction behind each roller. This design isolates the sample from the moving parts of the pump and allows for easy cleaning by removal and replacement of the flexible tubing. Unfortunately, peristaltic pumps are generally capable of only providing relatively low yields. They are, therefore, not well suited for

well evacuation in large diameter wells with high yields. However, they may be well suited for well evacuation in small diameter wells with low yields.

In the procedure for peristaltic pumps, ground water is withdrawn through a precleaned 6 mm I.D. teflon tube and discharged into a clean, calibrated one-liter heavy-wall erlenmeyer flask with a peristaltic pump on the outlet side of the sampling flask. Tubing includes 6 mm I.D. teflon, 6 mm I.D. tygon, and 6 mm O.D. glass.

The sampled water only contacts teflon and precleaned glass, and is then carefully transferred to appropriate glass sample containers for shipment to the laboratory. This system shall not be used for samples to be analyzed for volatile organics, because of the possibility of stripping volatile constituents from the sample under the reduced pressure occurring in these systems. It may, however, be used for non-volatile constituents such as metals or other organics.

### 5.3 Gas Lift Pumps

Gas lift pumps operate by releasing compressed gas through an air pipe inside a larger diameter discharge or eductor pipe. Mixing gas bubbles with the water results in a lower water density inside the discharge pipe, allowing the water to be blown to the surface. Numerous adaptations to the basic method of applying gas pressure to a water well and forcing a water sample out the discharge tube are available for use in monitoring applications. These include the use of a high-pressure hand pump and any reasonably flexible tubing. This provides a highly portable sampling unit. Similarly, a small air compressor or tank of compressed gas and the appropriate piping or flexible tubing can be used. Unfortunately, gas lift methods aerate samples and water

in the well, greatly limiting their suitability for both sample collection and well evacuation.

In general, gas pressure lift systems should not be used for sample collection as they have been shown to cause considerable changes in the groundwater character.

In gas lift pumps, compressed nitrogen is regulated into an approximate 1/2-inch diameter nylon tube connected to the sampler in the well. Groundwater that has permeated the sampler is forced up through an approximate 1/4-inch concentric nylon tube and sampled. The pressure and flow of nitrogen will be controlled with the regulator in order that the groundwater sample exits slowly and with a minimum of "spurting" at the end. One volume of groundwater will be flushed out and discarded to clean the tubes and obtain newly permeated groundwater. All sample bottles will be filled directly from the 1/4-inch tubing.

#### 5.4 Submersible Pumps

A variety of positive displacement pumps are available for use in withdrawing water from wells. These methods use some pumping mechanism placed in the well that forces water from the bottom of the well to the surface by some means of positive displacement. This minimizes the potential for aerating or stripping volatile organics from the sample during removal from the well.

The submersible centrifugal pump is one common example of a positive displacement pump. It works in a manner similar to the centrifugal suction lift pump previously described except that the pump and electric motor are lowered into the well. As the impeller rotates and fluid is brought into the pump, fluid is displaced up the transmission line and out of the well. These

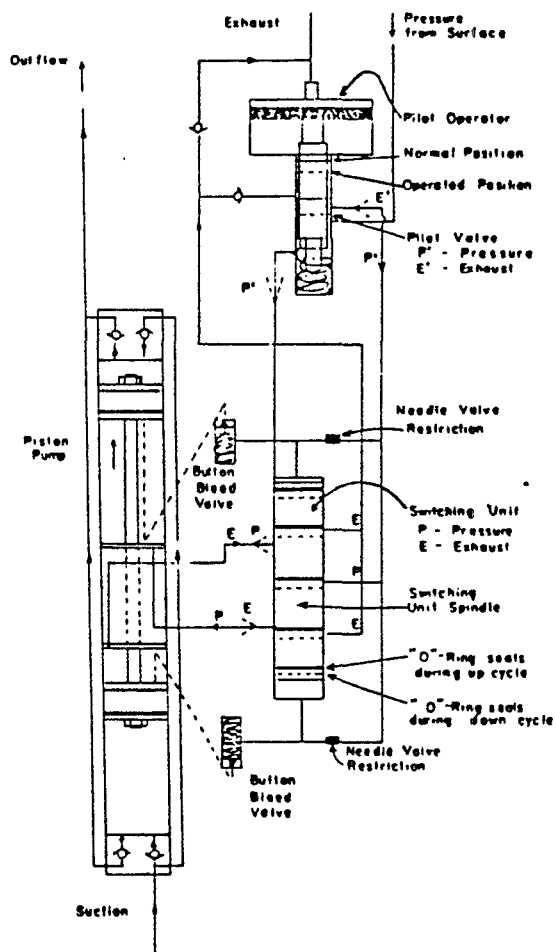
pumps are capable of providing a high yield. However, they require an external source of power and are frequently constructed with materials and contain lubricants incompatible with sampling certain constituents, particularly organics. They require considerable equipment and effort to move from well to well. The chief drawback, however, is the difficulty of avoiding cross contamination between wells. These systems are generally too expensive to allow for several separate units and field decontamination is very difficult. Decontamination should properly require solvents, which may lead to sample contamination. Use of submersible pumps, therefore, in multiple well programs, should be carefully considered against bailers.

#### 5.5 Piston Pumps

Piston-driven or reciprocating piston pumps are another example of common positive displacement pumps (see Figure 3). These pumps consist of a piston in a submerged cylinder operated by a rod connected to the drive mechanism at the surface. A flap valve or ball-check valve is located immediately above or below the piston cylinder. As the piston is lowered in the cylinder, the check valve opens, and water fills the chamber. On the upstroke, the check valve closes, and water is forced out of the cylinder up into the transmission line and to the surface. The transmission line or piston contains a second check valve that closes on the down stroke, preventing water from re-entering the cylinder. These pumps are capable of providing high yields. However, moving these pumps from well to well is difficult, and their use in monitoring programs may require that a pump be dedicated to each well. Many of these pumps are not constructed with materials compatible with monitoring certain constituents.

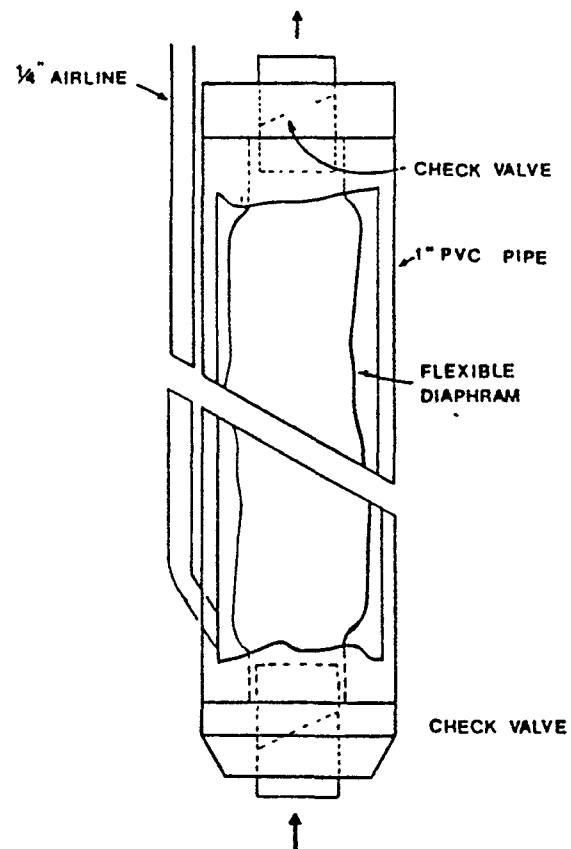
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Figure 3 - Gas-Driven Piston Pump



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Figure 4 - Gas-Operated Squeeze Pump



A special adaptation of this pump has recently become available for use in ground-water monitoring. These piston pumps use compressed gas rather than a rod connected to a driving mechanism at the surface to drive the pistons. This provides a much more convenient and portable means for collecting samples from Figures 3/4 monitoring wells. They provide good yields and can be constructed with materials compatible to many sampling programs. This pump is especially useful for sampling water with trace and volatile organic constituents.

#### 5.6 Gas-Operated Squeeze Pumps

Another positive displacement pump applicable for monitoring purposes is the gas-operated squeeze pump (see Figure 4). This pump was originally developed by R. F. Middelburg of the U.S. Geological Survey and consequently is referred to as the Middelburg pump. It consists principally of a collapsible membrane inside a long rigid housing, a compressed gas supply, and appropriate control valves. When the pump is submerged, water enters the collapsible membrane through the bottom check valve. After the membrane has filled, gas pressure is applied to the annular space between the rigid housing and membrane, forcing the water upward through a sampling tube. When the pressure is released, the top check valve prevents the sample from flowing back down the discharge line, and water from the well again enters the pump through the bottom check valve.

Gas-operated squeeze pumps offer a number of advantages for use in groundwater monitoring programs. They can be constructed in diameters as small as 1-in. and from a wide variety of materials. They are also relatively portable and are capable of providing a fair range of pumping rates. Most important, the driving gas

does not contact the water sample, thus eliminating possible contamination or gas stripping. However, they do require a gas source, and withdrawal of water from substantial depths may require large gas volumes and long pumping cycles.

#### 5.7 Jet Pumps

Jet pumps are a common submersible pump used in small domestic water wells and may in some cases be suggested for use in monitoring wells. These pumps operate by injecting water through a pipe down into the well. A venturi device is located at the intake portion of the pump. As the water injected from the surface passes through the constricted portion of the venturi, the velocity increases and pressure decreases according to Bernoulli's principle. If the discharge velocity at the nozzle is great enough, the pressure at this point will be lowered sufficiently to allow water to be drawn into the venturi assembly through the intake and brought to the surface with the original water injected into the well. This additional increment of water is then made available at the surface as the pump's output. Jet pumps generally require priming with water, and the water taken from the well mixes with water circulating in the system. A simple modification can be added to this design to circulate a portion of the pumped water back through the system, eventually reducing the concentration of primary water effectively to zero. This type pump is not suitable for sampling water for volatile organics since the turbulence produced by the venturi system will strip volatiles from the water.

#### 6.0 CHOOSING THE BEST METHOD

Since the nature of the pollutant or parameter being monitored is the primary factor for specifying well evacuation and sampling methods,



evaluation of any proposed method is most conveniently based on the general class of pollutant or parameter that requires monitoring in a particular program. For this purpose, pollutants and monitoring parameters can be categorized into the following classes: physical properties, metals, nonmetallic inorganics, general organic parameters and organics.

#### 6.1 Physical Properties

Physical properties include such parameters as conductance, color, pH, temperature, and turbidity. In general, most sampling methods are acceptable for these parameters, provided they allow a thorough rinsing between sampling events. However, methods that affect gas composition of the sample will affect pH. Consequently, the sampler should not use gas lift methods for sampling when pH is a parameter of interest. Since gas lift methods may also leave water in the well in a disturbed and aerated condition, these methods are not suitable for well evacuation when pH is a parameter of interest.

#### 6.2 Metals

Similarly, since concentration of metals can be significantly influenced by changes in pH, sampling for metals should not be allowed with gas lift or suction methods. The guidance provided above for well evacuation when pH is the parameter of interest also applies for metals. In addition, equipment used for monitoring metal concentrations should be metal free. Consequently, bailers and positive displacement pumps are most suitable for sampling metals, provided they are constructed of appropriate materials. The methods acceptable for well

evacuation are less restricted, but gas lift methods or equipment that alters the metal concentration of water remaining in the well through leaching or absorption should be avoided.

### 6.3 Inorganic, Nonmetallic Constituents

Inorganic, nonmetallic constituents or parameters include acidity, alkalinity, bromide, chloride, fluoride, nitrogen, etc. Most of the sampling and well evacuation methods described above are generally acceptable when considering the inorganic, nonmetallic parameters. However, for parameters affected by pH or dissolved gas changes, such as alkalinity, methods that minimize changes in dissolved gas composition are recommended for sampling. These methods include bailers, squeeze pumps, and piston pumps; gas lift techniques are not recommended for well evacuation.

### 6.4 Organics

Generalized organic parameters include parameters such as oil and grease, COD, TOC, and TOX. Most sampling and well evacuation methods are suitable for these parameters, with the exception of the more sensitive parameters such as TOX. These sensitive parameters require methods suitable for sampling volatile organic.

It has generally been recommended that sampling for volatile organics be done with a glass or Teflon bailer after flushing with a nonaerating pump. However, positive displacement pumps may also be acceptable in sampling, provided they are constructed with suitable materials (Teflon, glass, or stainless steel in most cases).

## 7.0 EXISTING IN-PLACE PUMPS

Occasionally ground water samples are obtained using wells which have existing in-place pumps. This limits the precautions the sampler can take to ensure a non-contaminated sample. Samples should be obtained from outlets as close as possible to the pump and should not be collected from leaky or faulty spigots or spigots that contain screens or aeration devices. The pump should be run for 5 - 10 minutes before the sample is collected, and the waste-water properly disposal of. A steady-flowing water stream at moderate pressure is desirable in order to prevent splashing and dislodging particles in the faucet or water line.

To collect the sample, remove the cap or stopper carefully from the sample bottle. Do not lay the bottle closure down or touch the inside of the closure. Avoid touching the inside of the bottle with your hands or the spigot. The sample bottle should not be rinsed out and it is not necessary to flame the spigot. The bottle should be filled directly to the top. The bottle closure and closure-covering should be replaced carefully and the bottle should be placed in a cooler (4 - 10°C) unless the sample is going to be processed immediately in the field.

## 8.0 SAMPLING FROM NATURAL SPRINGS

For springs in unconsolidated deposits, drive a well point or a slotted pipe one meter or less into the ground adjacent to the spring. Collect from the artesian flow or use a pump. Use plastic pipe and plastic well screens for trace metal determinations. To sample larger upwelling springs, attach the pump intake to a pole and submerge it in the mouth of the spring.

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## 9.0 REFERENCES

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## SURFACE WATER SAMPLING GUIDELINES

### 1.0 OBJECTIVE

The objective of obtaining surface water samples is to determine the surface water quality entering, leaving or affected by the site. Surface water samples are considered environmental samples (containing low concentrations of contaminants) but accurately quantifying the contaminants present is of utmost importance. In order to obtain a representative sample, the hydraulics of the water course must be determined before sampling, so that the most representative sample can be obtained.

Either grab or composite samples may be collected. Grab samples are collected at one particular point, and time. Flow- or time-weighted composite samples are composed of more than one aliquot collected at various sampling sites and/or at different times. Because of the unknown safety risks, as well as the changes in chemical nature of the sample that may occur through compositing, samples containing hazardous materials at significant concentrations shall not be composited. Environmental samples containing low levels of toxics may be composited.

If it is necessary to wade into the water course to obtain a sample, the team member shall be careful not to disturb bottom sediments and shall enter the water course downstream of the sampling location. While in reality, it is difficult to prevent disturbance of the sediments, it is the responsibility of the team member to minimize the introduction of sediments into the sample. If necessary, the sampling technician shall wait for the sediments to settle before taking the sample.

If the water course is moving, a depth integrated grab sample shall be obtained. A depth integrated sample is collected by lowering an open container against the flow, to a depth just above bottom. The container is then turned into the flow and raised at a rate that allows it to just fill when it reaches the surface. The sample shall be collected in the middle of the stream.

## 2.0 EQUIPMENT

The best choice of sampling equipment depends on the particular conditions at the site and the water body being sampled. The types of samplers available are:

- o Open tube
- o Pond Sampler
- o Manual Hand Pump
- o Weighted Bottle Sampler
- o Kemmerer Sampler
- o Extended Bottle Sampler.

Of these, the pond sampler and the weighted bottle sampler will be used most often. The criteria for selecting a sample collector are:

- o Disposable and/or easily decontaminated - A collection device may not be used again without sufficient cleaning.
- o Inexpensive - This is a necessity if the item is to be disposed of.
- o Ease of operation - Cumbersome safety clothing and the use of protective respiratory equipment dictate the use of simple tools.
- o Nonreactive - The device must not react with the sample in such a manner as to contaminate it.
- o Safe - The unit must not present a safety threat to the user.

### 3.0 SAMPLING METHODS

#### 3.1 Pond Sampler

The pond or dip sampler (Figure 1) consists of a container attached to the end of a long pole by an adjustable clamp. The pole can be of any non-reactive material such as wood, plastic or metal, as it will not be in contact with the sample itself. The sample shall be collected in a jar or beaker made of stainless steel, glass or non-reactive plastic. Preferably, a disposable beaker which can be replaced, shall be used at each station. Liquid wastes from water courses, ponds, pits, lagoons or open vessels are "ladled" into a sample container.

#### 3.2 Manual Hand Pumps

Manual pumps are available in various sizes and configurations. Manual hand pumps are commonly operated by peristaltic, bellows or diaphragm, and siphon action. Manual hand pumps which operate by a bellows or diaphragm, and siphon action should not be used to collect samples which will be analyzed for volatile organics.

The pump is operated according to manufacturer's instructions. The sample inlet hose is inserted into the liquid to be sampled, and a crank or bellows is activated.

To avoid contamination of the pump, a liquid trap consisting of a vacuum flask is inserted at the sample inlet hose to collect the sample (Figure 2). Teflon tubing shall be used for the inlet hose in order to avoid sample contamination. The hose and trap must be flushed between stations with a minimum of three volumes of liquid, and cleaned or replaced at the end of each day.

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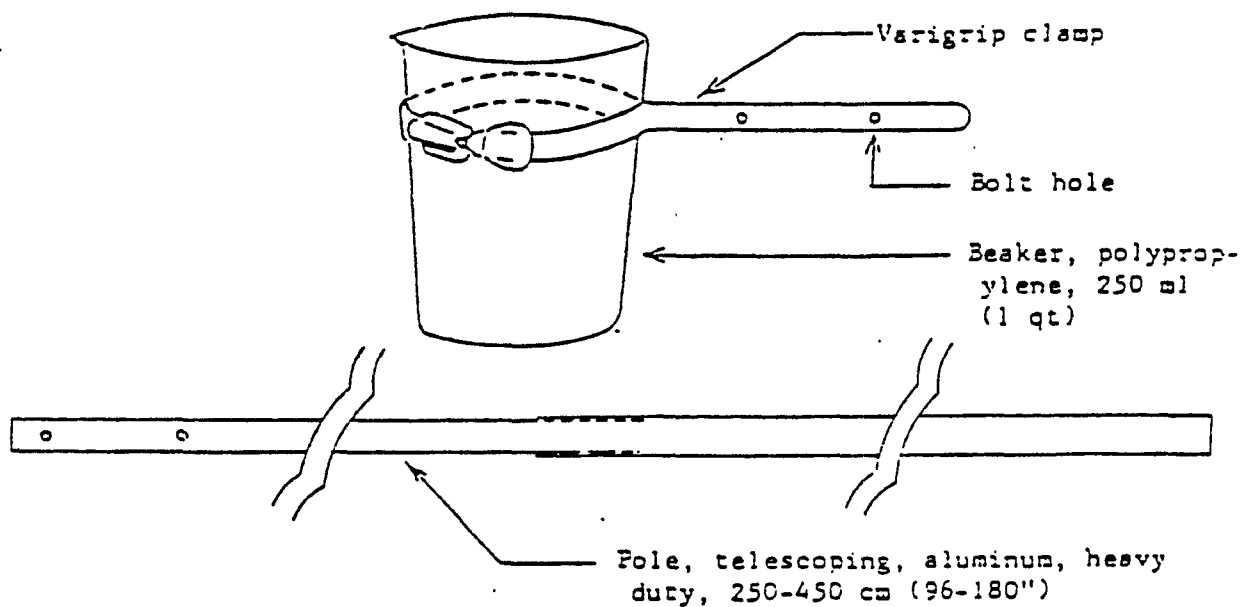


Figure 1 Pond sampler



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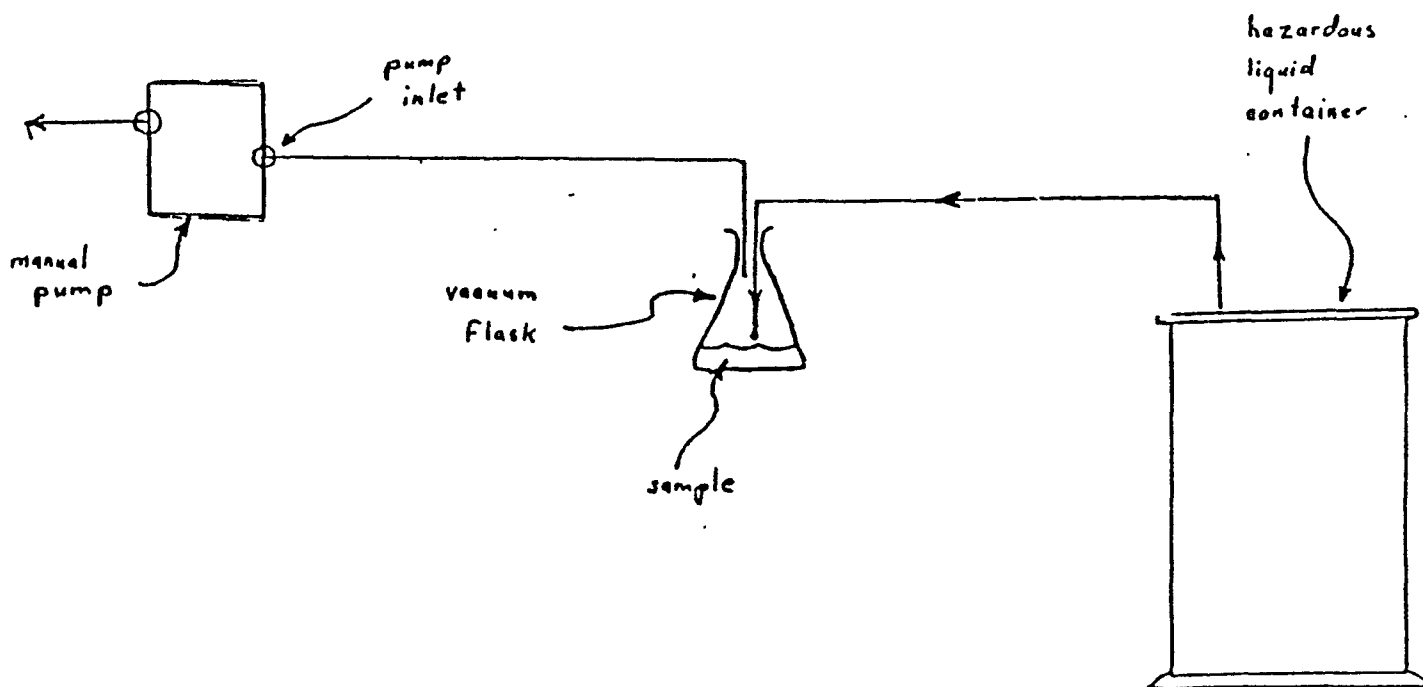


Figure 2

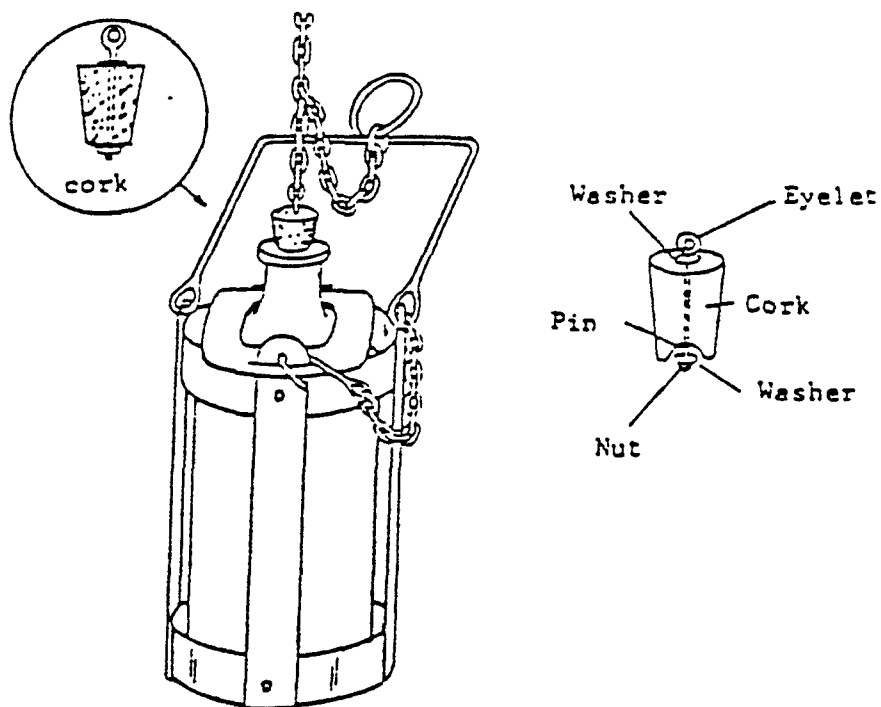
### 3.3 Weighted Bottle Sampler

The sampler (Figure 3) consists of a glass bottle, a weight sinker, a bottle stopper, and a line that is used to open the bottle and to lower and raise the sampler during sampling. There are variations of this sampler, as illustrated in the ASTM methods D 270 and E 300. This sampler can be either fabricated or purchased. Procedure for use:

1. Assemble the weighted bottle sampler as shown in Figure 3.
2. Gently lower the sampler to the desired depth so as not to remove the stopper prematurely.
3. Pull out the stopper with a sharp jerk of the sampler line.
4. Allow the bottle to fill completely, as evidenced by the cessation of air bubbles.
5. Raise the sampler and cap the bottle.
6. Wipe the bottle clean. The bottle can be used as the sample container.

Alternatives to the weighted bottle sampler are the Kemmerer sampler and the extended bottle sampler.

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1000-ml (1-quart) weighted  
bottle catcher

Figure 3 Weighted bottle sampler.

## PROCEDURE FOR USE AND MAINTENANCE OF FIELD NOTEBOOKS

### 1.0 INTRODUCTION

Field notebooks provide means for recording all data collecting activities performed at a site. As such, entries should be as descriptive and detailed as possible, so that a particular situation could be reconstructed without reliance on the collector's memory.

### 2.0 NOTEBOOK USE

Field notebooks shall be bound, 4 x 7 to 8 x 10.5 inch books with consecutively numbered pages. Notebooks shall be permanently assigned to field personnel, but are to be stored in site project files when not in use. Each notebook is identified by a document control number which indicates:

ZJX - FN - XXXX - XX

(Site Number - Field Notebook - Owner Identification - Sequence Number)

The cover of each notebook contains the following information:

- o Person or Organization to whom the book is assigned
- o Book Number
- o Site Name and Number
- o Start Date
- o End Date

Entries into the logbook may contain a variety of information. At the beginning of each entry the following information is recorded; the

date, start time, weather, all field personnel present, level of personal protection being used on-site, and the signature of the person making the entry should be noted.

All measurements made and samples collected are recorded. All entries should be made in pen. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialled. Entries should be organized into easily understandable tables if possible. A sample format is shown in Exhibit 1.

At each station where a sample is collected or a measurement made, a detailed description of the location of the station, which includes compass and rangefinder measurements, are recorded. The film roll number and number of photographs taken at the station are also noted.

All equipment used to make measurements is identified, including the date on which the equipment was calibrated.

Samples are to be collected following sampling procedures described in this manual. The equipment used to collect samples should be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. In addition, the identification of the container number into which the sample is placed in the field is recorded. Sample numbers are assigned prior to going on-site. Duplicates, which receive an entirely separate sample number, are noted under sample description. Significant field notebook entries (samples collected, significant observations) must be countersigned by another member of the project team.

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EXHIBIT 1  
TYPICAL FIELD NOTEBOOK ENTRY FORMAT

START TIME

DATE

WEATHER: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

FIELD PERSONNEL: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

LEVEL OF PERSONAL PROTECTION: \_\_\_\_\_

RECORDER'S SIGNATURE: \_\_\_\_\_  
\_\_\_\_\_

EQUIPMENT (NAME/CONTROL NO.): \_\_\_\_\_

CALIBRATION DATE: \_\_\_\_\_

Station No./Location Description: \_\_\_\_\_  
\_\_\_\_\_

Film Roll Number: \_\_\_\_\_ Photograph Numbers: \_\_\_\_\_

Station No.	Parameter (Units)
_____	_____
_____	_____
_____	_____

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Typical Field Notebook Entry Format (Cont.)

Date  
Start Time

Sampling Equipment: \_\_\_\_\_

No.	Time	Sample Description	Depth	Number	Volume	Chest No.	Comments
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[illegible]

## SAMPLE CLASSIFICATION, HANDLING AND SHIPMENT

### 1.0 INTRODUCTION

The protocols for the classification, handling, and shipment of samples collected on and adjacent to uncontrolled hazardous waste sites are presented in this procedure. Steps in the procedure should be followed to ensure the integrity of the samples, as well as protecting the welfare of the persons involved in the shipment, the shipper, and receiver of the samples. When sent by common carrier, the packaging, labeling and shipping of hazardous wastes and substances is regulated by the U.S. Department of Transportation (DOT; 49 CFR).

### 2.0 SAMPLE CLASSIFICATION

Samples obtained at uncontrolled hazardous waste sites are classified as either environmental samples or hazardous samples. Environmental samples are those which contain low levels of contaminants and require implementation of limited precautionary procedures. Hazardous samples are those which could possibly contain dangerous levels of contaminants. Hazardous samples must be packaged and labeled according to procedures specified by the U.S. DOT, or the state DOT, whichever is more stringent.

#### 2.1 Environmental Samples

Environmental samples are those samples known not to contain dangerously high levels of contaminants. If any doubt exists as to the extent of contamination, samples should be treated as hazardous.



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Environmental samples are collected in an appropriate container allowing approximately 10 percent air space (ullage) so that the container is not full at 130° F. If a head space is not desired for a particular analysis (i.e., volatile organic analyses), the container should be placed inside another container, to provide the desired head space. The sealed and labeled container is then placed inside a ziplock polyethylene bag which is also sealed. The sealed package is then placed inside a shipping container, packed so as to prevent breakage. No precautionary notices are required on the package exterior.

## 2.2 Hazardous Samples

Samples not designated as environmental samples or which are known to contain hazardous materials must be considered hazardous. DOT has established a prioritized system of transportation categories which depends on the degree of hazardousness of the material. The relevant portion of this listing is shown in Table 1.

Initially, all samples should be surveyed for radiation. If radiation levels are below 0.5 millirems per hour at the surface of the package material, the sample is not shipped as radioactive. If the radiation level exceeds 0.5 millirems per hour, the sample is shipped as radioactive. The Code of Federal Regulations Title 49, sub part I, should be consulted to provide proper shipping containment for shipping of a radioactive sample.

Poison "A" is the next category on the DOT list. Poison "A" substances (listed in Table 2) are defined by DOT as extremely dangerous poisonous gases or liquids of such toxicity that a very small amount of gas, or vapor of the liquid, mixed with air is life threatening. Many of the Poison "A" materials are gases or

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TABLE 1 DOT HAZARDOUS MATERIALS CLASSIFICATION

1. Radioactive Material	12. Combustible liquid (in containers having capacity exceeding 110 gallons)
2. Poison "A"	13. ORM-B
3. Flammable Gas	14. ORM-A
4. Non-flammable gas	15. Combustible liquid (in containers having capacity of 110 gallons or less)
5. Flammable liquid	16. ORM-E
6. Oxidizer	
7. Flammable Solid	
8. Corrosive Material (liquid)	
9. Poison B	
10. Corrosive Material (solid)	
11. Irritating Materials	

TABLE 2 CLASS "A" POISONS AND THEIR PHYSICAL STATE AT ROOM TEMPERATURES

<u>Compound</u>	<u>Physical State</u>
arsine	gas
bromoacetone	liquid
chloropicrin & methyl chloride mixture	gas
chloropicrin & non-flammable, non-liquified compressed gas mixture	gas
cyanogen chloride	gas at temperature greater than 13.1 degrees C
cyanogen gas	gas
gas identification set	
gelatin dynamite (H.E. Germaine)	-
grenade (with poison "A" gas charge)	-
hexaethyl tetraphosphate & compressed gas mixture	gas
hydrocyanic acid (prussic) solution	liquid
hydrocyanic acid, liquified	gas
insecticide liquified gas, containing poison "A" or poison "B" material	gas
methyldichloroarsine	liquid
nitric oxide	gas
nitrogen peroxide	gas
nitrogen tetroxide	gas
nitrogen dioxide, liquid	gas
parathion & compressed gas mixture	gas
phosgene (diphosgene)	liquid

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compressed gases and would not be found in drum type containers. Liquid poison "A"'s would probably be found in closed containers which provides a "worst case" situation. Not all samples found in drums should be labeled Poison "A". Based upon the information available, a judgement must be made as to the hazard class of the sample. If the sample is suspected or determined to fall within the Poison "A" classification, packaging procedures specified by DOT should be followed as specified in 49 CFR, Part 173.326-328.

The next two classifications in the DOT series are "flammable" or "non-flammable" gases. Few, if any, gas samples are expected to be collected at uncontrolled hazardous waste sites. Use this category only when shipping containerized gases or gas samples.

The next category to be considered is "flammable liquids". Hazardous samples in liquid form, unless known to fall into a lower category, will be handled, packaged and shipped at this level of concern. However, lesser categories will generally not be considered because flashpoint testing required to drop to a lower level is difficult and possibly dangerous in the field. It is more practical to handle samples at the "flammable" level than to undertake field determination of the flash point.

Solids samples known or suspected to be flammable are shipped as flammable solids. Non-flammable solids may be shipped as Poison "B", corrosive, or irritant materials based on characteristics of the sample and DOT regulations.

Small quantities (i.e., less than 5 pounds of a solid and one pint of liquid in a single package) of hazardous waste samples can be shipped under the Other Regulated Material (ORM) category. If the material is known, the ORM-A or ORM-B classification may be used. 49 CFR 173 parts K and L list the ORM A and ORM B

Materials. If the material is unknown or not listed under parts K and L, the ORM-E category may be used. The ORM-E category is for hazardous waste liquids and solids, not otherwise specified.

The following steps apply to handling flammable liquid and solid samples.

### 3.0 SAMPLE PACKAGING

Samples are collected in glass containers with non-metallic, teflon-lined screw caps. Sufficient ullage (approximately 10 percent by volume) is allowed so that the container is not liquid-full at 130 degrees Fahrenheit. If an air space in the inner most container cannot be tolerated in order to maintain sample integrity, the sample shall be placed within a second container to provide the required air space.

In collecting a solid material, the container plus contents shall not exceed 1 pound net weight. Large quantities of material, up to 1 gallon, may be collected if the flash point of the sample can be determined to be 73 degrees Fahrenheit or higher. If this is the case, this information should be marked on the outside container (carton, etc.), but only a single (1 gallon or less) bottle may be packed in an outside container with 10 percent air space. The shipping papers are required to state that the "flash point" is 73 degrees or higher.

Seal the sample container and place each in a separate 2-mil thick (or thicker) ziplock polyethylene bag. The sample identification tag should be positioned to enable it to be read through the bag.

Each sealed bag shall be placed inside an appropriate sized metal can or other DOT approved container with enough noncombustible, absorbent,

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cushioning material (e.g., bentonite, vermiculite or diatomaceous earth) to prevent breakage and provide for absorption of liquid; one bag per can. Pressure close the can and use clips, tape or other positive means to hold the lid securely, tightly, and effectively.

The metal cans or other DOT-approved container, or a single 1-gallon bottle shall be placed into a strong outside container, such as a metal picnic cooler or an approved fiberboard box and surrounded with noncombustible, absorbing packaging material for stability during transport.

#### 4.0 MARKING AND LABELING

Abbreviations are used only where specified by DOT. The following information shall be placed on each metal can, other DOT-approved container, or 1-gallon bottle. Appropriate labels are supplied by the Regional Equipment Manager.

The label should contain the laboratory name and address and appropriate DOT hazardous shipment category. As a conservative approach "Flammable Liquid N.O.S. UN1922" can be used for most liquids and "Flammable Solid N.O.S. UN1325" for most solids. If you know for certain that the sample is not a flammable liquid or solid, then another category in the DOT hierarchy should be used. Not other specified (N.O.S.) is used when the sample is not identified. Identify the sample by name and UN identifier when known.

The following DOT labels shall be placed on the outside of the can (or bottle), depending on contents.

"Cargo Aircraft Only" (Danger Peligro) - "Flammable Liquid",  
"Flammable Solid", "Dangerous When Wet" or "Corrosive".

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If the cans are placed in an exterior container, both the container and inside can (or bottle) must have the same markings and labels as above.

"LABORATORY SAMPLES" and "THIS SIDE UP" or "THIS END UP" should also be marked on the top and/or front side of the outside container, and upward pointing arrows should be placed on all 4 sides of the exterior container.

#### 5.0 SHIPPING PAPERS

Abbreviations shall be used only where specified below. The bill of lading supplied by the carrier should be completed and the certification statement signed (if not provided by the carrier, standard industry form shall be used) with the following information in the order listed. One form may be used for more than one exterior container.

"Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325", "Cargo Aircraft Only", "Limited Quantity" or "Ltd. Qty.", "Laboratory Samples", "Net Weight \_\_\_\_\_" or "Net Volume \_\_\_\_\_" of hazardous contents, by item, if more than one metal can is inside of exterior container.

The net weight or net volume must be placed just before or just after the "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." description.

A complete chain-of-custody record, enclosed in an envelope is included in the sample container.

Containers must be loaded or otherwise sealed.

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#### 6.0 TRANSPORTATION

All samples should be shipped by Federal Express. "Cargo Only" aircraft may be used, but hazardous samples shall not be transported by any carrier which also carries passengers.

Hazardous or environmental samples may be transported by CDM personnel in private vehicles.

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## SAMPLE IDENTIFICATION PROCEDURE

### 1.0 INTRODUCTION

A coding system will be used to identify each sample taken during the sampling program. This coding system will provide a tracking record to allow retrieval of information about a particular sample and assure that each sample is uniquely identified.

### 2.0 SAMPLE IDENTIFICATION

Each sample is identified by a unique code which indicates the site number, sample type, sample point, and sequence number. An example of the sample identification code will be as follows:

150-SW-010-003  
123-SW-001-001

Where 150 indicates the site number, SW the sample type, 010 the sample point, and 003 the sequence number.

A three digit number will be used to identify each site. The site number will be obtained from the National Project Management Office and be used as an identifier for all samples collected at that site.

A two letter designation is used to identify the specific type of sample being taken followed by three digits which indicate the station location. The sample types which will be collected during the remedial site investigations are:

SW - Surface water grab sample (streams, rivers, lakes, runoff)

SF - Surface water flow (continuous measurement)



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GW - Ground water sampled from various types of wells

WQ - Continuous water quality measurement

SL - Soil samples (beds, surface soil, surface borings)

SS - Subsurface samples (drill core, split spoon, etc.)

SD - Sediment samples collected from stream beds, lagoons, etc.

LG - Lagoon samples

TS - Tank samples including above ground and below ground enclosures

DM - Drum samples

AM - Meteorological station

AG - Gaseous air samples

AP - Particulate air samples

AO - Organic air samples

MS - Trace metal samples

RS - Rock samples

BI - Biological samples

A three digit number will be used to identify a sample point location.  
This location can be a soil sample point, bore hole, well, drum, tank

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surface water sample point, lagoon point, air monitor station, or any other point where a source material, water, soil, core, or air sample may be taken.

The final sample identification code will be a sequence identifier. This number will be used to identify separate samples collected at the same sample point.

The sample type, sample point identifier and sequence number codes should be established for each sample to be collected prior to field activities. This step can be performed manually or through the use of computer programs.

## CHAIN-OF-CUSTODY

### 1.0 INTRODUCTION

Chain-of-Custody procedures provide documentation of the handling of each sample from the time it is collected until it is destroyed. Such a written record is especially important if the results of analyses of samples will be used to support litigation.

### 2.0 CHAIN-OF-CUSTODY PROTOCOLS

To maintain a record of (1) sample collection, (2) transfer of sample between personnel, (3) sample shipment, and (4) receipt by the laboratory which will analyze the sample (which will then continue the chain-of-custody within their laboratory records), a "Chain-of-Custody Record" is filled out for each sample type at each sampling location. Form F6260, or a resonable facsimile, will be used on the REM II program. Each time the samples are transferred to another custodian, signatures of the person relinquishing the sample and receiving the sample, as well as the time and date, should document the transfer. As stated in Procedure 5622004 each sample container will be labeled with a pressure sensitive gummed label. The label contains the sample number, date and time of sample collection, location of sample collection, depth of sample collection, preservatives used and the names of collector(s) and initial(s).

The chain-of-custody form (F6260) will include four pressure sensitive copies so that four forms are filled out simultaneously. The On-site Coordinator retains the original and any extra copies, and additional copies are shipped with the samples until they are received by the laboratory(ies). If samples are split to different labs, a copy will go to each lab. Care must be taken that all four copies are legible. If additional duplicate sheets are required, the person relinquishing the samples is responsible for filling out additional copies, or making reproductions. The original must be returned by the On-site Coordinator to the site project files.

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The Chain-of-Custody Record will be placed in a ziplock bag and placed inside of all shipping and transport containers. All samples will be shipped by Federal Express to the laboratory specified in the project operations plan. Samples should be packed so that no breakage will occur. The shipping or external container should be sealed with evidence tape and initialled so that any sign of tampering is easily visible.

## SAMPLE BOTTLE PREPARATION, SAMPLE PRESERVATION AND MAXIMUM HOLD TIMES

### 1.0 INTRODUCTION

Complete and unequivocal preservation of samples, either domestic sewage, industrial wastes, or natural waters, is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent is not likely to be achieved. At best, preservation techniques can retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. Degradation of the sample ceases only if it is preserved at a temperature of absolute zero ( $-273^{\circ}\text{C}$ ). Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low  $\mu\text{g/l}$  range.

Methods of preservation are relatively limited and are intended generally to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing.

The recommended preservative for various constituents is given in Table 1. Preservation techniques for samples requiring more than simple refrigeration or filtering are discussed in section 3.0. Other information provided in the table is an estimation of the volume of sample required for the analysis, the suggested type of container, and the maximum recommended holding times for samples properly preserved.

To minimize changes in the chemical quality of a sample during shipping and storage prior to analysis, the sampler should use proper containers and adequate procedures for sample preservation and

Parameter No./name	Container <sup>1</sup>	Preservation <sup>1,2</sup>	Maximum holding time <sup>4</sup>
13. All and total	P G	Cool 4°C, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	6 hours.
14. Tests	P G	do	Do
15. Chemical oxygen demand	P G	Cool 4°C	14 days.
16. Bromide	P G	do	Do.
17. Biochemical oxygen demand carbonaceous	P G	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
18. Chemical oxygen demand	P G	Cool 4°C	48 hours.
19. Chloride	P G	None required	28 days.
20. Chlorine total residual	P G	Cool 4°C	48 hours.
21. Color	P G	do	Do
22-24. Cyanide total and amenable to chlorination	P G	Cool 4°C NaOH to pH>12, 0.6g ascorbic acid <sup>8</sup>	Analyze immediately
25. Fluoride	P G	do	48 hours.
26. Hardness	P G	None required	14 days. <sup>6</sup>
27. Hydrogen ion (pH)	P G	HNO <sub>3</sub> to pH<2 H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
28. Kjeldahl and organic nitrogen	P G	None required	6 months.
29. Metals <sup>1</sup>	P G	None required	Analyze immediately
30. Chromium VI	P G	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
31. Mercury	P G	do	24 hours.
32-34. 5-8 10 12 13 19 20 22 26 29 30 32-34 36 37 45 47 51 52, 58-60 62 63 70-72, 74 75 Metals except chromium VI and mercury	P G	do	28 days.
35. Nitrate	P G	do	6 months.
36. Nitrate-nitrite	P G	Cool 4°C	48 hours.
37. Nitrite	P G	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
38. Oil and grease	P G	Cool 4°C	48 hours.
39. Organic carbon	P G	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
40. Orthophosphate	P G	Cool 4°C HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	Do
41. Oxygen Dissolved Probe	P G	Filter immediately Cool 4°C	48 hours.
42. Winkler	G Bottle and top	None required	Analyze immediately
43. Phenols	do	Fix on site and store in dark	8 hours.
44. Phosphorus (elemental)	G only	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
45. Phosphorus total	G	Cool 4°C	48 hours.
46. Residue total	P G	Cool 4°C H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days.
47. Residue Filterable	P G	Cool 4°C	7 days.
48. Residue Nonfilterable (TSS)	P G	do	48 hours.
49. Residue Settlesable	P G	do	7 days.
50. Residue volatile	P G	do	48 hours.
51. Silica	P	do	7 days.
52. Specific conductance	P G	do	28 days.
53. Sulfate	P G	do	Do
54. Sulfide	P G	Cool 4°C add zinc acetate plus sodium hydroxide to pH>9	Do
55. Sulfite	P G	None required	7 days.
56. Surfactants	P G	Cool 4°C	Analyze immediately
57. Temperature	P G	do	48 hours.
58. Turbidity	P G	None required	Analyze
59. Table IC—Organic Tests <sup>6</sup>	P G	Cool, 4°C	48 hours.
60. 13 18-20 22 24-26 34-37 39-43 45-47 56 66 86 89 92-95 97	G Teflon-lined septum	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	14 days.
61. Purgeable Halocarbons	do	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup> HCl to pH2 <sup>10</sup>	Do
62. 6 57 90 Purgeable aromatic hydrocarbons	do	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup> Adjust pH to 4-5 <sup>10</sup>	Do
63. 3 4 Acroline and acrylonitrile	G Teflon-lined cap	Cool, 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	7 days until extraction,
64. 23 30 44 49 53 67 70 71 83 85 96 Phenols <sup>11</sup>	do	do	40 days after extraction
65. 7 38 Benzodioxines <sup>11</sup>	do	do	7 days until extraction <sup>13</sup>
66. 14 17 48 50-52, Phthalate esters <sup>11</sup>	do	Cool 4°C	7 days until extraction
67. 72-74 Nitrosamines <sup>12,14</sup>	do	Cool 4°C store in dark, 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	40 days after extraction.
68. 76-82 PCBs <sup>11</sup> acrylonitrile	do	Cool 4°C	Do
69. 54 55 65 69 Nitrosaromatics and isophorone <sup>11</sup>	do	Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup> store in dark	Do
70. 1 2 5 8-12 32, 33 58 59 64 66 84 86 Polynuclear aromatic hydrocarbons <sup>11</sup>	do	do	Do
71. 15 16 21 31 75 Halothanes <sup>11</sup>	do	Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	Do
72. 29 35-37 60-63 91 Chlorinated hydrocarbons <sup>11</sup>	do	Cool 4°C	Do
73. 87 TCDD <sup>11</sup>	do	Cool 4°C 0.008% Na <sub>2</sub> S <sub>2</sub> O <sub>5</sub> <sup>6</sup>	Do
74. Table ID—Pesticides Tests	do	Cool, 4°C pH 5-9 <sup>15</sup>	Do
75. 1-70 Pesticides <sup>11</sup>	do	do	Do
76. Table IE—Radiological Tests	P G	HNO <sub>3</sub> to pH<2	6 months.
77. 1-5 Alpha, beta and radium	do	do	do

<sup>1</sup> Polyethylene (P) or Glass (G)

## TABLE 1 NOTES

<sup>2</sup> Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

<sup>3</sup> When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.95 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.82 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

<sup>4</sup> Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee or monitoring laboratory has data on file to show that the specific types of samples under study are stable for the longer time and has received a variance from the Regional Administrator under § 136.3(a). Some samples may not be stable for the maximum time period given in the table. A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

<sup>5</sup> Should only be used in the presence of residual chlorine.

<sup>6</sup> Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

<sup>7</sup> Samples should be filtered immediately on-site before adding preservative for dissolved metals.

<sup>8</sup> Guidance applies to samples to be analyzed by GC/LC or GC/MS for specific compounds.

<sup>9</sup> Sample receiving no pH adjustment must be analyzed within seven days of sampling.

<sup>10</sup> The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

<sup>11</sup> When the extractable analytes of concern fall within a single chemical category the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories the sample may be preserved by cooling to 4°C reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 5-9. Samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to the optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re the analysis of benzidine).

<sup>12</sup> If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

<sup>13</sup> Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxygen free) atmosphere.

<sup>14</sup> For the analysis of diphenylhydrosamine, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub><sup>6</sup> and adjust pH to 7-10 with NaOH within 24 hours of sampling.

<sup>15</sup> The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>.

shipment. In many programs, the sampler will need to split the sample into several different samples, specifying different sample containers and preservation methods for each sample based on the analyses of interest.

## 2.0 SAMPLE CONTAINERS

Selection of sample containers should be based on the analytical parameters of interest. The containers should be made of materials that are nonreactive. Glass and polyethylene containers are the most commonly accepted, and both may be used when sampling many constituents. When metals are the analytes of interest, however, polyethylene containers with polypropylene caps are preferred. When organics are the analytes of interest, glass containers with teflon-lined caps must be used. When choosing containers for an individual monitoring program, the sampler should refer to the selected analytical method being used. The documentation of analytical procedures generally includes a listing of acceptable containers.

Depending on the analyses to be performed and the nature of the samples being collected, the sample container must be treated according to specific procedures. Bottles should be washed as described in 'general bottle washing' if: (1) they will be stored for later (not specified) usage, (2) they will be used for composite samples for a variety of routine analyses, (3) they will be used for routine analyses not requiring special preparation.

### 2.1 General Bottle Cleaning

#### 2.1.1 Bottle Material

Polypropylene, glass, Teflon

#### 2.1.2 Bottle Size

Dependent upon determinatioons required.

#### 2.1.3 Cleaning Reagents

1. Phosphate-free detergent
2. Distilled water
3. Reagent-grade methanol

#### 2.1.4 Procedure

1. Rinse bottles with tap water.
2. Soak bottles in detergent solution for approximately thirty (30) minutes.
3. Scrub bottles with a brush.
4. Rinse bottles several times with tap water to remove the detergent.
5. Rinse bottles thoroughly, several times, with distilled water.
6. Rinse bottles with methanol.
7. Bake for one hour at 300', or air dry.

### 2.2 Bottle Cleaning for Metals Determination

#### 2.2.1 Bottle Material

Polypropylene, borosilicate glass, or Teflon

#### 2.2.2 Bottle Size

200-500 ml

#### 2.2.3 Cleaning Reagents

1. Detergents
2. 1:1 Nitric acid



3. 1:1 Hydrochloric acid
4. Distilled water

#### 2.2.4 Procedure

1. Follow general bottle cleaning procedure.
2. Add 1:1 nitric acid to bottles, cap, and shake briefly.
3. Allow bottles to stand for approximately 30 minutes, shaking them intermittently.
4. Pour acid from bottles and rinse them with tap water.
5. Repeat steps 2, 3, and 4 with 1:1 hydrochloric acid.
6. Rinse bottles thoroughly, several times, with deionized distilled water.

Note 1: Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water, and that the last traces of chromium are nearly impossible to remove. This is especially important if chromium is to be included in the analytical scheme. A commercial product - NOCHROMIX - available from Godax Laboratories, 6 Varick Street, New York, N.Y. 10013, may be used in place of chromic acid. [Chromic acid should not be used with plastic bottles.]

Note 2: If it can be documented through an active analytical quality control program using spiked samples, reagent and sample blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

#### 2.3 Bottle Cleaning for Oil and Grease Determinations

2.3.1 Bottle Material

Glass with Teflon-lined cap

2.3.2 Bottle Size

One (1) liter

2.3.3 Cleaning Reagents

1. Acid solution of 1+1 nitric acid
2. Detergent
3. Distilled water
4. Freon-113

2.3.4 Procedure

1. Follow general bottle cleaning procedure.
2. Rinse bottles (excluding caps) with acid solution.
3. Pour acid from bottles and rinse them with tap water.
4. Rinse bottles thoroughly, several times, with distilled water.
5. Rinse bottles with Freon-113.

2.4 Bottle Cleaning for Extractable Organics

2.4.1 Bottle Material

Glass with Teflon-lined cap.

2.4.2 Bottle Size

One (1) gallon

#### 2.4.3 Cleaning Reagents

1. Detergent
2. Distilled water
3. Pesticide grade hexane

#### 2.4.4 Procedure

1. Follow general bottle cleaning procedure.
2. Rinse bottles several times with distilled water.
3. Rinse bottles and caps two times with pesticide-quality hexane.

### 3.0 PRESERVATION TECHNIQUES

Samples should be preserved at low temperatures in the dark during transport to the laboratory for analysis. Appropriate chemical preservation should be performed in the field for various analytical parameters at the time of sampling. Regardless of the method of preservation, analyses should be performed as soon after sampling as is practicably possible in accordance with EPA and Standard Methods holding times.

Methods of sample preservation are relatively limited and are generally intended to (1) retard biological action, (2) retard hydrolysis of chemical compounds and complexes, (3) reduce volatility of constituents, and (4) reduce absorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration, and freezing. A summary of sample preservation measures is presented in the table below. When selecting preservation techniques for a specific monitoring program, the sampler should again refer to the guidance provided in the documentation of the analytical methods to be used. In some instances, the optimal method for sample preservation may be inappropriate owing to the restrictions placed on

the transport of certain chemicals by shippers. When shipping restrictions prevent the use of some reagents for sample preservation, the most appropriate and permissible technique should be used. Frequently, this will require refrigeration of the sample during transit.

In general, all environmental water samples should be placed in an insulated ice chest, and iced to lower the sample water temperatures to 4°C or less.

Environmental samples are generally dilute (in terms of pollutant concentration) samples taken in an area surrounding a spill or dump site.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, etc. and require special handling procedures because of their potential toxicity or hazard. These samples should not be preserved in any way, including refrigeration.

### 3.1 Preservation Techniques for Metals

Before collection of the sample a decision must be made as to the type of data desired, i.e., dissolved, suspended, total or total recoverable. For container preference, maximum holding time and sample preservation at time of collection see Table 1. Drinking water samples containing suspended and settleable material should be prepared using the total recoverable metal procedure.

#### 3.1.1 Dissolved

For the determination of dissolved constituents the sample must be filtered through a 0.45 u membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus using plain, non-grid marked, membrane filters are recommended to avoid possible contamination.)

### Summary of Sample Preservation Methods

Preservative	Action	Application
HgCl <sub>2</sub>	Bacterial Inhibitor	Nitrogen forms, phosphorus forms
Acid (HNO <sub>3</sub> )	Metals solvent, prevents precipitation	Metals
Acid (H <sub>2</sub> SO <sub>4</sub> )	Bacterial inhibitor	Organic samples (COD, oil, and grease, organic carbon)
	Salt formulation with organic bases	Ammonia, amines
Alkali (NaOH)	Salt formulation with volatile compounds	Cyanides, organic acids
Refrigeration	Bacterial inhibitor	Acidity-alkalinity, organic materials, BOD, color, odor, organic N, carbon, etc., biological organism (coliform, etc.)

Use the first 50-100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled HNO<sub>3</sub> to a pH of <2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the sample (see Note). If hexavalent chromium is to be included in the analytical scheme, a portion of the filtrate should be transferred before acidification to a separate container and analyzed as soon as possible. Analyses performed on a sample so treated shall be reported as "dissolved" concentrations.

Note: If a precipitate is formed upon acidification, the filtrate should be digested using 3.1.3. As much as 25

ml of conc. HCl/liter, may be required to stabilize certain types of highly buffered samples if they are to be stored for any length of time. Therefore, special precautions should be observed for preservation and storage of unusual samples intended for metal analysis.

#### 3.1.2 Suspended

For the determination of suspended metals a representative volume of unpreserved sample must be filtered through a 0.45  $\mu$  membrane filter. When considerable suspended material is present, as little as 100 ml of a well mixed sample is filtered.

#### 3.1.3 Total

For the determination of total metals the sample is acidified with 1:1 redistilled  $\text{HNO}_3$  to a pH of less than 2 at the time of collection. The sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. If much suspended material is present, as little as 50-100 ml of well mixed sample will most probably be sufficient. (The sample volume required may also vary proportionally with the number of metals to be determined.)

#### 3.1.4 Total Recoverable

To determine total recoverable metals, acidify the entire sample at the time of collection with conc. redistilled  $\text{HNO}_3$ , 5 ml/l.

### 3.2 Preservation Technique for Cyanide

### 3.2.1 Chemical Preservative

NaOH

Ascorbic acid (used only in presence of residual chlorine)

### 3.2.2 Procedure

1. Add 2 ml of 10 N NaOH per liter of sample. This should increase the pH to greater than 12.
2. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator or in an ice chest filled with water and ice to maintain temperature at 4°C.
3. Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

## 3.3 Preservation Techniques for Nitrogen

### 3.3.1 Ammonia

Add 2 ml conc.  $H_2SO_4$  per liter and cool to 4°C.

### 3.3.2 Kjeldahl, Total

Samples may be preserved by addition of 2 ml of conc.  $H_2SO_4$  per liter and stored at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Preserved samples should be analyzed as soon as possible.

### 3.3.3 Nitrate Plus Nitrite, Nitrate

Analysis should be made as possible. If analysis can be made within 24 hours, the sample should be preserved by refrigeration at 4°C. When samples must be stored for more than 24 hours, they should be preserved with sulfuric acid (2 ml  $H_2SO_4$  per liter) and refrigeration.

Caution: Samples for reduction column must not be preserved with mercuric chloride.

### 3.3.4 Nitrite

Samples should be analyzed as soon as possible. They may be stored for 24 to 48 hours at 4°C.

## 3.4 Preservation Technique for Dissolved Oxygen (Winkler)

3.4.1 Where possible, collect the sample in a 300 ml BOD incubation bottle. Special precautions are required to avoid entrainment or solution of atmospheric oxygen or loss of dissolved oxygen.

3.4.2 Where samples are collected from shallow depths (less than 5 feet), use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples collected from depths of greater than 5 feet.

3.4.3 When a Kemmerer sampler is used, the BOD sample bottle should be filled to overflowing (overflow for approximately 10 seconds). Outlet tube of Kemmerer should be inserted to bottom of BOD bottle. Care must be taken to prevent turbulence and the formation of bubbles when filling bottle.

3.4.4 At time of sampling, the sample temperature should be recorded as precisely as required.



3.4.5 Do not delay the determination of dissolved oxygen in samples having an appreciable iodine demand or containing ferrous iron. If samples must be preserved either method below may be employed.

3.4.5.1 Add 2 ml of manganous sulfate solution and then 2 ml of alkaline iodide-azide solution to the sample contained in the BOD bottle. Both reagents must be added well below the surface of the liquid. Stopper the bottle immediately and mix the contents thoroughly. The sample should be stored at the temperature of the collection water, or water sealed and kept at a temperature of 10 to 20°C, in the dark.

3.4.5.2 Add 0.7 ml of conc.  $\text{H}_2\text{SO}_4$  and 1 ml sodium azide solution (2 g  $\text{NaN}_3$  in 100 ml distilled water) to sample in the BOD bottle. Store sample as above.

3.4.6 If either preservation technique is employed, complete the analysis within 4-8 hours after sampling.

### 3.5 Preservation Technique for Phosphorus

1. If benthic deposits are present in the area being sampled, great care should be taken not to include these deposits.
2. Sample containers may be of plastic material, such as cubitainers, or of Pyrex glass.
3. If the analysis cannot be performed the same day of collection, the sample should be preserved by the addition of 2 ml conc.  $\text{H}_2\text{SO}_4$  per liter and refrigeration at 4°C.

### 3.6 Preservation Technique for Sulphide

1. Samples must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently

added to the sample may convert the sulfide to an unmeasurable form.

2. If the sample is not preserved with zinc acetate and NaOH, the analysis must be started immediately. Similarly, the measurement of dissolved sulfides must also be commenced immediately.

### 3.7 Preservation Techniques for Organics

#### 3.7.1 Chemical Oxygen Demand

1. Collect the samples only in glass bottles with teflon-lined caps.
2. Biologically active samples should be tested as soon as possible.
3. The collection of a composite sample and/or division of the sample into separate aliquots is not generally possible due to losses on equipment and imperfect mixing.
4. Samples should be preserved with sulfuric acid to a pH <2 and maintained at 4°C until analysis.

#### 3.7.2 Oil and Grease; Petroleum Hydrocarbons

1. A representative sample of 1 liter volume should be collected in a glass bottle. Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. The entire sample is consumed by this test; no other analysis may be performed using aliquots of the sample.
2. A delay between sampling and analysis of greater than 4 hours requires sample preservation by the addition of 5 ml HCl. A delay greater than 48 hours also requires refrigeration for sample preservation.

#### 3.7.3 Organic Carbon

1. Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the

samples. Note: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

2. Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. Also, samples should be kept cool (4°C) and protected from sunlight and atmospheric oxygen.
3. In instances where analysis cannot be performed within two hours (2 hours) from time of sampling, the sample is acidified ( $\text{pH} \leq 2$ ) with HCl or  $\text{H}_2\text{SO}_4$ .

#### 3.7.4 Phenolics

1. Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. The sample should be kept at 4°C and analyzed within 24 hours after collection.

#### 4.0 REFERENCE

- U.S. EPA, 1983. Methods for the chemical analysis of water and wastes. March 1983. EPA-600/4-79-020.
- A.P.H.A. 1975. Standard methods for the examination of water and wastewater. 14th ed.
- U.S. EPA, 1983. Test methods for evaluating solid waste. SW-846.
- U.S. EPA, 1983. RCRA Permit Writer's Manual: Ground-Water Protection (40 CFR Part 264, Subpart F), Geotrans Inc., EPA Contract No. 68-01-6464.
- USEPA/NWWA, 1981. Manual of Ground-Water Sampling Procedures, NWWA/EPA Series.
- U.S. EPA, 1984. 40 CFR Part 136. Federal Register Part VIII. October 26, 1984.

## OPERATION PROCEDURE LUDLUM MODEL 3-5 SURVEY METER

### 1.0 INTRODUCTION

#### 1.1 Instrument Description

The Ludlum Model 3-5 Survey Meter is a waterproof portable alpha, beta, gamma survey instrument that operates on two standard "D" cell flashlight batteries. The instrument features a regulated high-voltage power supply adjustable from 400 to 1,500 volts. It provides a 4-linear range from 0-20 mR/hr. The instrument includes a unimorph speaker with an ON-OFF capability. The instrument also features a fast-slow meter response, meter reset button, meter light pushbutton and a 6-position switch for selecting battery check or scale in multiples of X0.1, X1, X10, and X100. Each range multiplier has its own calibration potentiometer.

#### 1.2 Description of Controls and Functions

1. The meter has a readout of 0 to 4K CPM and 0 to 2 mR/hr for actual count of 0 to 400,000 CPM and 0 to 200 mR/hr.
2. Range Calibration Adjustments are located on the instrument face recessed with each multiplier position.
3. The Range Multiplier Selector Switch is a 6-position switch marked OFF, BAT, X100, X10, X1, X0.1. Turning the range selector switch from the OFF to BAT position provides a check of battery level. The remaining positions select one of the ranges of operating. Multiply the scale reading by the multiplier for determining the actual reading.
4. The High Voltage Adjustment provides a means to vary the high voltage from 400 to 1,500 volts. The high voltage setting may be checked at the connector with an appropriate voltmeter.

5. The RES button, when depressed, provides a rapid means to drive the meter to zero.
6. The LAMP pushbutton, when depressed, lights the meter face.
7. The AUDIO ON-OFF switch operates the unimorph speaker. The frequency of the clicks is relative to the rate of the incoming pulses. The higher the rate is, the higher the audio frequency. The audio should be turned off when not required to minimize battery drain.
8. The Fast-Slow (F/S) toggle switch selects meter response. In the F position, the meter indicates 90% of the final meter reading in five seconds. In the S position, 90% of the final meter reading is reached in 11 seconds. In "F" position, there is a fast response and large meter deviation. In the "S" position, there is a slow response and damped meter deviation.

### 1.3 Detectors

The instrument is supplied with three detectors, Models 44-6 and 44-9 detectors, and Model 44-3 Scintillation counter.

The Model 44-3 Scintillation counter is a highly sensitive detector used to detect beta and gamma radiation. (Additional details and specifications will be added when received from Ludlum.)

The Model 44-6 detector is a low sensitivity detector also used for monitoring beta and gamma radiation. (Additional details and specifications will be added when received from Ludlum.)

The Model 44-9 detector is an overall survey detector used for monitoring of alpha, beta, and gamma radiation. (Additional details and specifications will be added when received from Ludlum.)

## 2.0 SPECIFICATIONS

Power: two standard "D" size batteries

Four Linear Powers: from 0 to 200 mR/hr; meter scale presentation - 0 to 2 mR/hr with range multiples of X0.1, X1, X10, X100.

Sensitivity: 40 millivolts (+20mV, -16mV)

Audio: built-in unimorph speaker with an ON-OFF switch

High Voltage: externally adjustable from 400 to 1,500 volts

Response: 5 or 25 seconds for 90% of final meter reading

Linearity: plus or minus 5% full scale

Calibration Stability: less than 15% variance to battery end point

Meter: 50 microamp, 2.5 inch scale, with pivot and jewel suspension

Connector: Series "C", 706 U/G

## 3.0 OPERATION

### 3.1 Instrument Setup

1. Select detector to be used to survey site. The instrument can use alpha/beta/gamma and beta/gamma detectors.
2. Connect the cable to the instrument and detector.
3. Turn the range switch to BAT. The meter should deflect to the battery check portion of the meter scale. If the meter does not respond, check batteries for condition, age, or proper polarity. Replace or reinsert batteries as necessary.

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As a check of battery function, press lamp button and check for a light on the meter.

4. Turn instrument range switch to X100. Expose the detector to a check source. The speaker should click with the AUDIO ON-OFF switched to ON.
5. Move the range switch to the lower scales until a meter reading is indicated.
6. Place F/S toggle in either fast or slow response position.
7. With the detector adjacent to the known source, depress the RES button. The meter should zero.

### 3.2 Field Use

Take measurements throughout the site giving emphasis to drums, lab packs, spill solid materials, and areas containing obvious spills of source material.

Screen all samples, cuttings and borings for radioactivity.

Record all meter settings in the field log book on an appropriate form.

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## EQUIPMENT AND INSTRUMENT CALIBRATION AND MAINTENANCE, GENERAL REQUIREMENTS

### 1.0 INTRODUCTION

The general guidelines for calibrating and maintaining instruments and monitoring equipment are presented in this document.

### 2.0 CALIBRATION AND MAINTENANCE PROCEDURES

Calibration and maintenance procedures are documented for each piece of equipment affecting quality. Calibration and maintenance procedures are developed based on manufacturer's specifications and are retained in the Site Investigation Procedures Manual. These procedures include, but are not limited to:

1. Equipment identification (name) and description.
2. Equipment specifications.
3. Calibration and/or maintenance schedule.
4. Equipment necessary to accomplish calibration (where applicable).
5. Procedure for calibration and/or maintenance.

### 3.0 CALIBRATION LABEL

Instruments requiring calibration and/or maintenance have a prominently displayed sticker containing the following information:

1. Date of calibration and/or maintenance.
2. Next due date for calibration and/or maintenance.
3. Initials of person performing calibration and/or maintenance.
4. Span gas and concentration(s) (if applicable).
5. Span or sensitivity setting (if applicable).



#### 4.0 EQUIPMENT LOG BOOK

An equipment log book is issued to record the life history of each measuring and testing device used in activities affecting quality. This book is a three ring binder in which individual records for each piece of equipment are maintained. A form such as F6101 or a reasonable facsimile should be used to maintain the calibration and maintenance record. The record should include:

1. Equipment identification (name) and control number.
2. Date of calibration and/or maintenance.
3. Condition of equipment.
4. Activity performed on instrument (calibration and/or maintenance).
5. Adjustments made and accuracy of equipment prior to and following calibration (where applicable).
6. Record of equipment failure or inability to meet specifications (where applicable).
7. Initials of person performing calibration/maintenance.
8. Next due date for calibration and/or maintenance.

#### 5.0 CALIBRATION/MAINTENANCE FORM

An instrument specific calibration/maintenance form will be developed to record data relating to each individual calibration/maintenance event. A single form will be used for each calibration/maintenance event. In addition to the data recorded in the calibration/maintenance log, the following items should also be included in the instrument specific form (where applicable).

1. Calibration calculations and curves.
2. Span gas type and concentrations.
3. Span or sensitivity range settings.
4. Specifics on repairs and parts replaced, added, or removed.

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5. Instrument's overall condition.

#### 6.0 FIELD CALIBRATION

As part of normal field operations, some instruments require calibration prior to, during, and/or after field use. This field operation calibration should remain separate from pre-field calibrations and should not be used as a substitute for standard calibration activities. Field calibration should be recorded in field log books or on field forms as part of the normal field data collection process. Field calibration records should not be included in the history log.

#### 7.0 INSTRUMENTS NOT IN COMPLIANCE

If the calibration schedule is not adequately maintained, or if accuracy as reported in specifications cannot be attained for a specific instrument, that instrument is labelled "HOLD" and is unavailable for use until it is repaired and specifications are attained.

## CALIBRATION PROCEDURE FOR THE HNu PI 101

### 1.0 INTRODUCTION

#### 1.1 Content

This procedure presents the steps required to calibrate the HNu Model PI 101 photoionization analyzer. This instrument should be calibrated after each field use or prior to each field use if the instrument has not been calibrated during the previous 14 calendar days. The principle of detection and operating procedures are described in Procedure 5607001. This procedure presents calibration steps only.

#### 1.2 Equipment

- o Calibration Gas (2 ranges)

Low range 0-20 ppm and mid range 20-200 ppm Isobutylene gas for standard field operation when contaminants are unknown or a mixture of gases is present. Isobutylene is the gas used for general calibration because of the instrument's relatively high sensitivity to it and the non-toxic nature of the gas.

Note: A specialty gas may be required if a single atmospheric contaminant is present and the contaminant has a sensitivity different from that of the calibration gas. See procedure for 5607001 for a discussion on specialty calibration.

- o Tubing and fittings (See Figure 1).
- o Rotometer or bubble flow meter.
- o Calibration Form F6264.
- o Table 1 for ionization potentials for compounds of interest.

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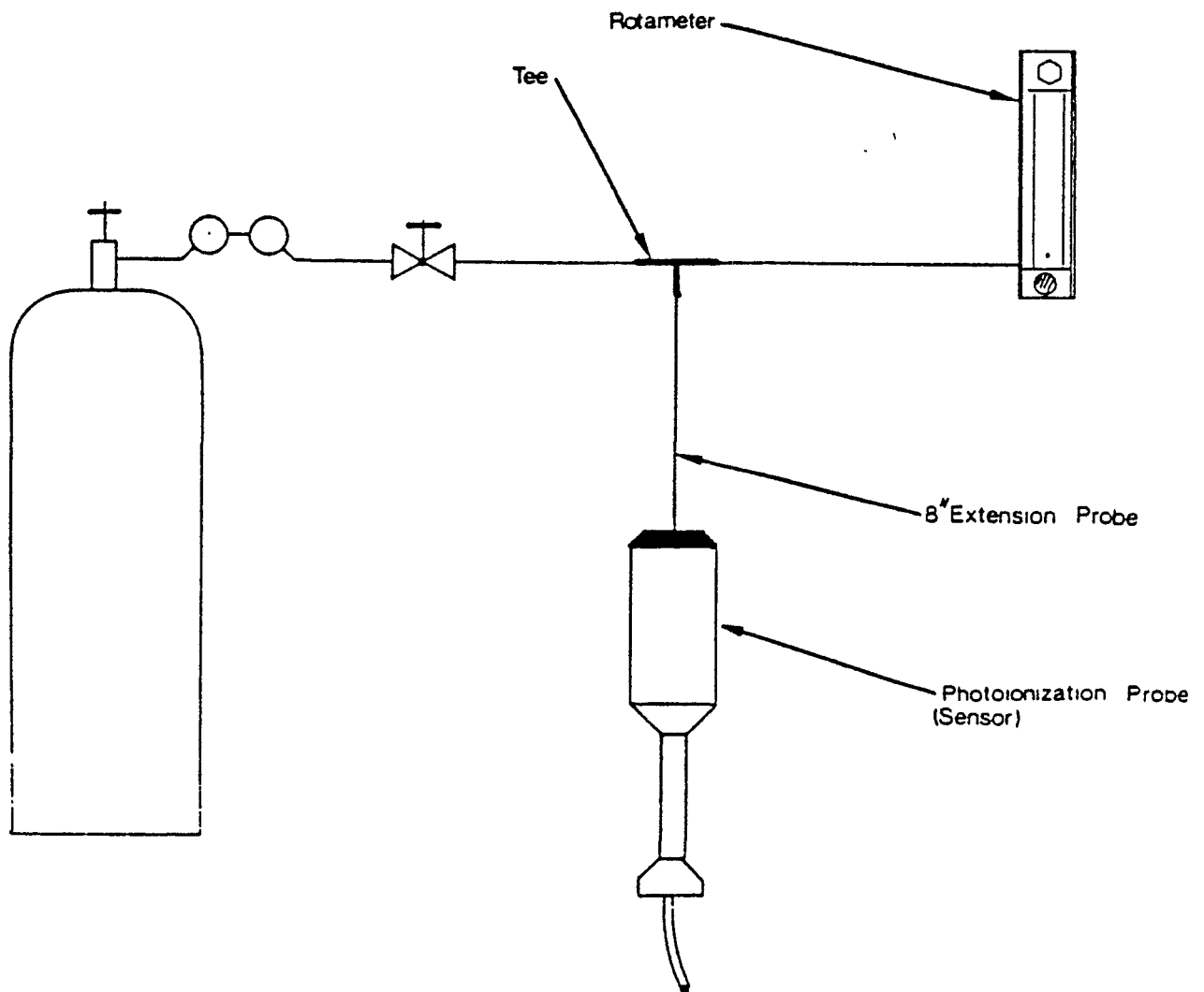


FIGURE 1 RECOMMENDED CALIBRATION PROCEDURE FOR PHOTOIONIZATION ANALYZER

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TABLE 1 RELATIVE SENSITIVITIES FOR VARIOUS GASES  
(10.2 eV Lamp)

Species	Photoionization Sensitivity*
p-xylene	11.4
m-xylene	11.2
benzene	10.0 (reference standard)
toluene	10.0
diethyl sulfide	10.0
diethyl amine	9.9
styrene	9.7
trichloroethylene	8.9
carbon disulfide	7.1
isobutylene	7.0
acetone	6.3
tetrahydrofuran	6.0
methyl ethyl ketone	5.7
methyl isobutyl ketone	5.7
cyclohexanone	5.1
naptha (85% aromatics)	5.0
vinyl chloride	5.0
methyl isocyanate	4.5
iodine	4.5
methyl mercaptan	4.3
dimethyl sulfide	4.3
allyl alcohol	4.2
propylene	4.0
mineral spirits	4.0
2,3-dichloropropene	4.0
cyclohexene	3.4
crotonaldehyde	3.1
acrolein	3.1
pyridine	3.0
hydrogen sulfide	2.8
ethylene dibromide	2.7
n-octane	2.5
acetaldehyde oxime	2.3
hexane	2.2
phosphine	2.0
heptane	1.7
allyl chloride (3-chloropropene)	1.5
ethylene	1.0
ethylene oxide	1.0
acetic anhydride	1.0
a-pinene	0.7
dibromochloropropane	0.7
epichlorohydrin	0.7
nitric oxide	0.6

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TABLE 1 RELATIVE SENSITIVITIES FOR VARIOUS GASES  
(10.2 eV Lamp) (Continued)

Species	Photoionization Sensitivity*
b-pinene	0.5
citral	0.5
ammonia	0.3
acetic acid	0.1
nitrogen dioxide	0.02
methane	0.0
acetylene	0.0

\*Expressed in ppm (v/v).

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## 2.0 CALIBRATION PROCEDURE

2.1 CDM employs a two-point standardization procedure to facilitate proper instrument calibration over the 0-20 ppm and 20-200 ppm operating ranges. Two distinct mixtures of the calibration gas (isobutylene) in air are used. Each mixture should give a 3/4 scale deflection in its respective operating range.

### 2.2 Instrument Setup.

2.2.1 Remove Instrument cover by pulling up on the side straps.

2.2.2 Prior to calibration, check the function switch (Figure 2) on the control panel to make sure it is in the OFF-positon. The probe nozzle, is stored inside the instrument cover. Remove cover plate by pulling up on the pins that fasten the cover plate.

2.2.3 Remove the nozzle from the cover. Assemble probe by screwing nozzle into casing.

2.2.4 Attach probe cable to instrument box by inserting 12 pin interface connector of the probe cable into the connector on the instrument panel. Match the alignment keys and insert connector. Turn connector in clockwise direction until a distinct snap and lock is felt.

2.2.5 Turn the function switch to the Battery Check position. When the battery is charged, the needle should read within or above the green battery arc on the scale plate. If the needle is below the green arc or the red LED light

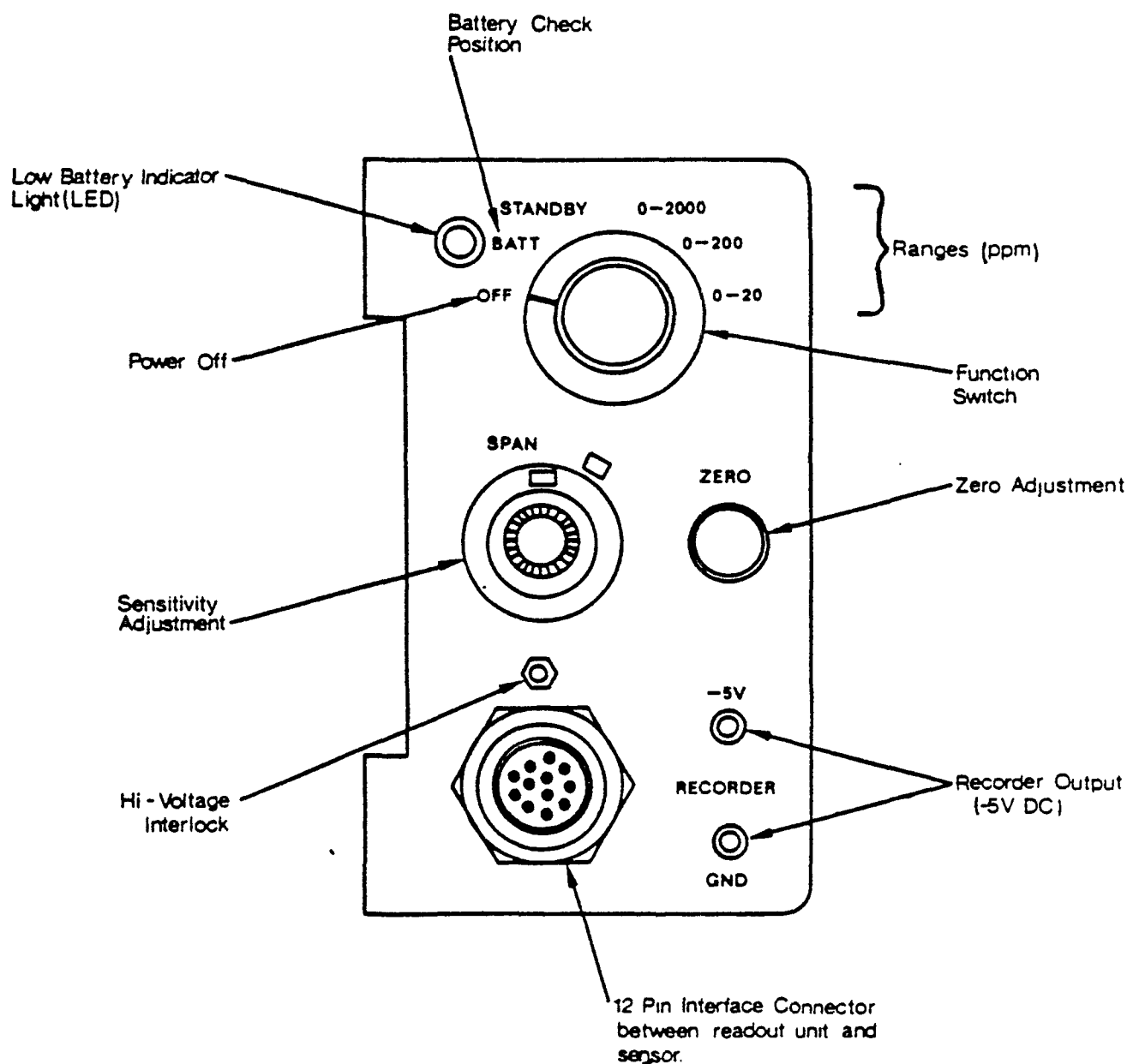


FIGURE 2 CONTROL PANEL FEATURES



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comes on, the instrument should be recharged prior to making any measurements. Implement step 3.0 to recharge battery.

- 2.2.6 Turn the function switch to the ON position. In this position, the UV light source should be on. To verify, gaze at the end of the probe for a purple glow. Do not look directly at the lamp itself. If the lamp does not come on refer to maintenance step 4.1.2.
- 2.2.7 To zero the instrument, turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counter clockwise rotation yields a downscale deflection. (Note: no zero gas is needed since this is an electronic zero adjustment.) If the span adjustment is changed during instrument calibration, the zero should be rechecked and adjusted. If necessary wait 15 to 20 seconds to ensure that the zero reading is stable. Readjust as necessary.

### 2.3 Calibration Steps

- 2.3.1 Insert one end of T tube (Figure 1) into probe. Insert second end of probe into calibration gas in the 20-200 ppm range. The third end of probe should have the rotometer (bubbler) attached.
- 2.3.2 Set the function switch in the 0-200 ppm range.

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- 2.3.3 Crack the valve on the pressured calibration gas container until a slight flow is indicated on the rotometer. The instrument will draw in the volume required for detection with the rotometer indicating excess flow.
- 2.3.4 Adjust the span potentiometer so that the instrument is reading the exact value of the calibration gas. (Calibration gas value is labeled on the cylinder).
- 2.3.5 Turn instrument switch to the standby position and check the electronic zero. Reset zero potentiometer as necessary following step 2.3.7.
- 2.3.6 Record on form F6264 all original and readjusted settings as specified in the form.
- 2.3.7 Next, set the function switch to the 0-20 ppm. Remove the mid range (20-200 ppm) calibration gas cylinder and attach the low range (0-20 ppm) calibration gas cylinder as described above.
- 2.3.8 Do not adjust the span potentiometer. The observed reading should be  $\pm$  3ppm of the concentration specified for the low range calibration gas. If this is not the case, recalibrate the mid range scale repeating procedures 3.3.1 to 3.2.7 above. If the low range reading consistently falls outside the recommended tolerance range, the probe light source window likely needs cleaning. Clean window following step 4.1.3. When the observed reading is within the required tolerances, the instrument is fully calibrated.

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2.3.9 Complete forms F6264 and F6265 for the respective instrument being calibrated.

### 3.0 BATTERY RECHARGING

3.1 Place plug on end of charger cable into jack on left side of instrument case

3.2 Plug charger into 120V AC supply.

3.3 To ensure that charger is functioning, turn the function switch to the battery check position. The meter should go upscale if the charger is working correctly and correctly inserted.

3.4 The battery is completely charged overnight (ca, 14 hours).

3.5 When disconnecting charger, remove from 120 V AC before removing mini phone plug.

### 4.0 TROUBLE SHOOTING AND MAINTENANCE

#### 4.1 General Fault Determination and Correction

4.1.1 Battery level is low. Recharge if necessary implementing step 3.0. If the battery will not recharge it will have to be replaced.

4.1.2 UV lamp function. Gaze at sample inlet when mode switch is on an instrument function position and observe for purple glow of lamp. If the lamp does not glow in any of the three instrument function positions, it may be burned out and will have to be replaced. To replace the lamp:

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1. Turn the function switch to the off position and disconnect the probe connector from the readout unit.
2. Remove the exhaust screw found near the base of the probe (Figure 3).
3. Grasp the end cap in one hand and the probe shell in the other and gently pull to separate the end cap and lamp housing from the shell.
4. Loosen the screws on the top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing. Care must be taken so that the ion chamber does not fall out of the end cap and the lamp does not slide out of the lamp housing.
5. Turn the end cap over in your hand and tap on the top of it; the ion chamber should fall out of it.
6. Place one hand over the top of the lamp housing and tilt slightly. The light source will slide out of the housing.
7. Replace lamp with one of same energy source as the one removed by sliding it into the housing. Note: the amplifier board and instrument circuitry are calibrated for one light energy source. Insertion of a lamp of a different energy level will produce false instrument readings.
8. Place the ion chamber on top of the lamp housing, checking to ensure that the contacts are aligned.
9. Place the end cap on top of the ion chamber and replace the two screws. The screws should be tightened only enough to seal the "O" ring. Do not overtighten.
10. Line up the pins on the base of the lamp housing with the pins inside the probe shell. Gently slide the housing assembly into the probe shell. Do not force the assembly as it only fits one way.
11. Replace and tighten the exhaust screw.
12. Reconnect the 12 pin connector and turn instrument mode switch to a function position. Check for glow of lamp. If lamp still does not function the instrument has an electrical short or other problem that will have to be corrected at the factory.

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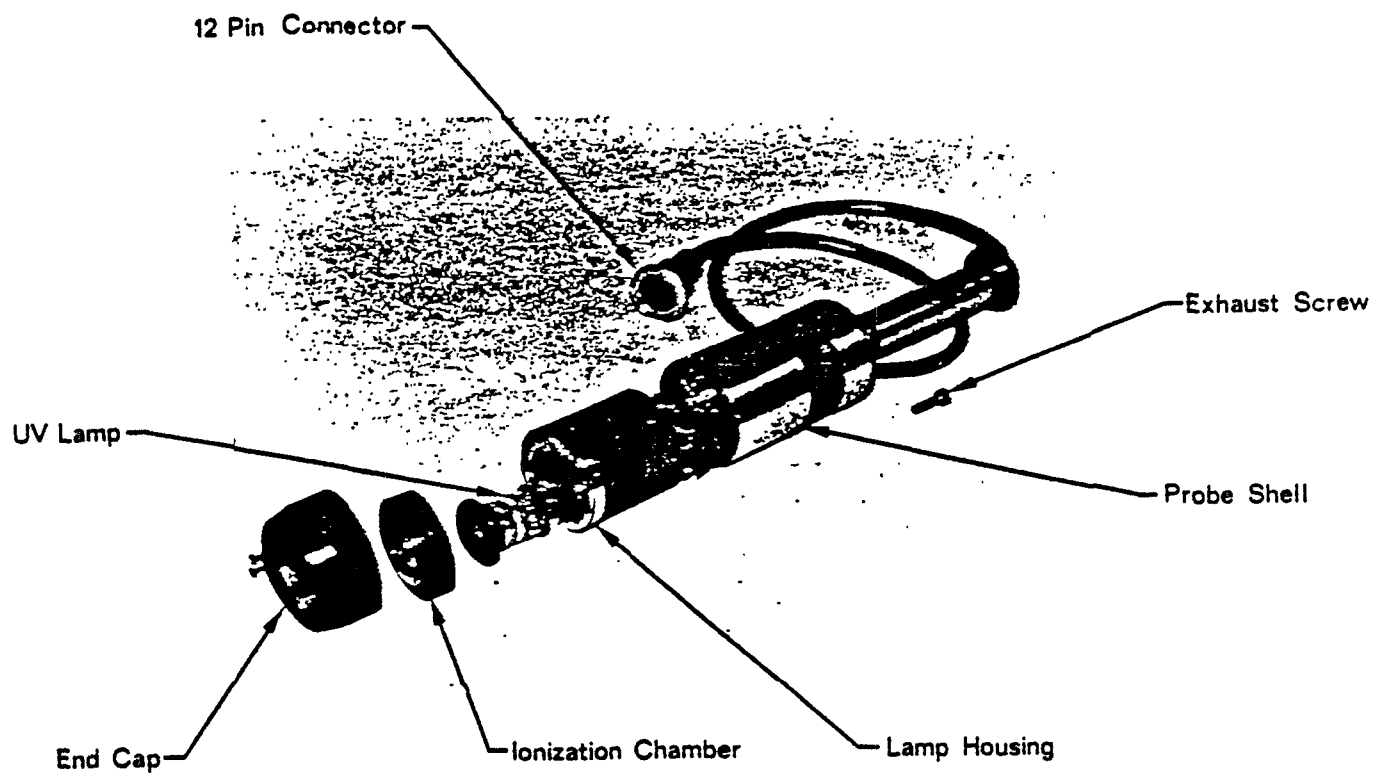


FIGURE 3 COMPONENT PARTS OF PROBE

4.1.3 Instrument appears to be functional, but responses are lower than expected or erratic. The window of the light source may be dirty and need to be cleaned. To clean the light source window:

1. Disassemble the probe assembly by repeating steps 1 through 6 under 4.1.2 above.
2. Clean the window of the light source using compound provided with instrument and soft clean cloth.  
Important: use cleaning compound on the window of the 10.2 eV lamp only. The cleaning compound may damage the windows of the 9.5 and 11.7 eV lamps.
3. Reassemble the probe assembly repeating step 7 through 12 above.

#### 4.2 Specific Faults

4.2.1 No meter response in any switch position (including BATT CHK)

1. Broken meter movement: Tip instrument rapidly from side to side. Meter needle should move freely, and return to zero.
2. Electrical connection to meter is broken: Check all wires leading to meter and clean the contacts of quick-disconnects.
3. Battery is completely dead: Disconnect battery and check voltage with a volt-ohm meter.
4. Check 2 amp fuse.
5. If none of the above solves the problem, consult the factory.

4.2.2 Meter responds in BATT CHK position, but reads zero or near zero for all others.

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1. Power supply defective: Check power supply voltages per Figure 4. If any voltage is out of specification, consult the factory.
2. Input transistor or amplifier has failed: Rotate zero control; meter should deflect up/down as control is turned. Open probe; both transistors should be fully seated in sockets.
3. Input signal connection broken in probe or readout: Check input connector on printed circuit board. Should be firmly pressed down. Check components on back side of printed circuit board. All connections should be solid, and no wires should touch any other object. Check all wires in readout for solid connections.

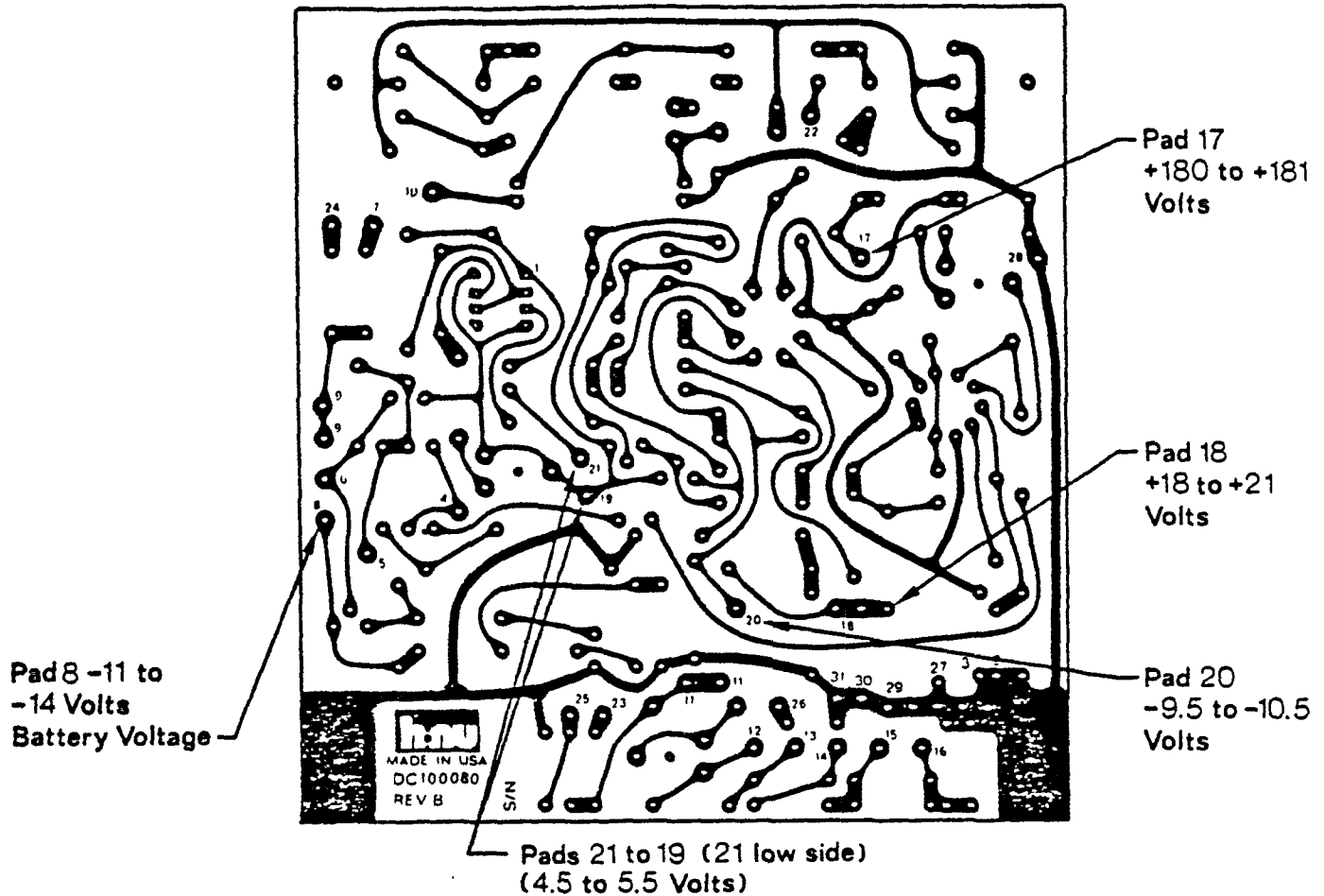
4.2.3 Instrument responds correctly in BATT CHK, and STBY, but not in measuring mode.

1. Check to see the light source is on (See Section 4.1.2).
2. Check high voltage power supply (see Figure 4).
3. Open end of probe, remove lamp and check high voltage on lamp contact ring.
4. If high voltage is present at all above points, light source has most likely failed. Consult the factory.

4.2.4 Instrument responds correctly in all positions, but signal is lower than expected.

1. Check span setting for correct value.
2. Clean window of light source (See 4.1.3).
3. Double check preparation of standards.
4. Check power supply 180 V output. See Figure 4.
5. Check for proper fan operation. Check fan voltage. See Figure 4.
6. Rotate span setting. Response should change if span pot is working properly.

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All Voltages Respect to Ground							
pads	voltage	pads	voltage	pads	voltage	pads	voltage
1	- 5.7 V	9	- 12.2V	17	180V	25	0
2	GRD	10	- 12.1V	18	+ 19.4V	26	0
3	GRD	11	0	19	- 10.6V	27	GRD
4	-10.7V	12	0	20	- 9.7V	28	0
5	- 11.3V	13	0	21	- 14.5V	29	GRD
6	- 12.1V	14	0	22	- 400V	30	GRD
7	0	15	0	23	0	31	GRD
8	- 12.2V	16	0	24	0		

Figure 4 Power Supply PC Board



4.2.5 Instrument responds in all switch positions, but is noisy (erratic meter movement).

1. Open circuit in feedback circuit. Consult the factory.
2. Open circuit in cable shield or probe shield. Consult the factory.

4.2.6 Instrument response is slow and/or irreproducible.

1. Fan operating improperly. Check fan voltage. See Figure 4.
2. Check calibration and operation.

4.2.7 Low battery indicator.

1. Indicator comes on if battery charge is low.
2. Indicator also comes on if ionization voltage is too high.

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CALIBRATION AND MAINTENANCE PROCEDURES  
CENTURY SYSTEMS PORTABLE ORGANIC VAPOR ANALYZER MODEL OVA-128

## 1.0 INTRODUCTION

This procedure presents steps required to calibrate and maintain the model OVA-128 organic vapor analyzer. Specifications and operating principles and procedures are presented in Procedure 5607003.

## 2.0 CALIBRATION

### 2.1 General

The OVA is capable of responding to nearly all organic compounds. However, the response will vary from compound to compound. The responses of some compounds relative to methane, are presented in Table 1. For precise analyses it is necessary to calibrate the instrument to a specific compound of interest, particularly if that compound contains elements other than carbon and hydrogen. For general use, the instrument is calibrated to methane.

Internal electronic adjustments are provided to calibrate and align the electronic circuits. There are four such adjustments, all located on the electronics board. One adjustment potentiometer, R-38, is factory set and is used to set the power supply voltage. Potentiometer R-38 thus should never be adjusted. The remaining three adjustments, R-31 (X1), R-32 (X10), and R-33 (X100) are used for setting the electronic amplifier gain for each of the three calibration ranges. Access to the adjustments is accomplished by removing the instrument from its case.

TABLE 1 - RESPONSE OF OVA TO HYDROCARBONS RELATIVE TO METHANE

Compound	Relative Response (%)
Methane	100 (Reference)
Propane	64
N-butane	61
N-pentane	100
Ethylene	85
Acetylene	200
Benzene	150
Tolvene	120
Ethane	90
Acetone	60
Methyl Ethyl Ketone	80
Methyl Isobutyl Ketone	100
Methyl Alcohol	15
Ethyl Alcohol	25
Isopropyl Alcohol	65
Carbon Tetrachloride	10
Chloroform	65
Trichloroethylene	70
Vinyl chloride	35

## 2.2 Methane Calibration

### 2.2.1 Equipment

- o Calibration gas (75-90 ppm methane)
- o T-tube assembly

### 2.2.2 Instrument Startup

Start instrument by:

1. Move PUMP switch to ON and check battery condition by moving the INSTR switch to the BATT position.

Recharge battery (section 3.0) if battery level is low.

2. Move INSTR switch to ON and allow 5 minutes for warmup.
3. Place instrument in vertical position and check flow rate.
4. Open the H<sub>2</sub> TANK VALVE and H<sub>2</sub> SUPPLY VALVE.
5. Depress Ignitor button for 6 seconds or until hydrogen ignites, whichever is shorter. If hydrogen fails to ignite, wait 2 minutes and attempt to ignite hydrogen.
6. Once lit, wait 5 minutes for instrument to stabilize before starting calibration procedure.
7. Open instrument cover to expose circuit board.

#### 2.2.3 Calibration

Calibration should be performed in a well ventilated area.

1. Set CALIBRATE switch to X10.
2. For methane calibration the GAS SELECT control should be set to 300. Check to ensure that this control is set at 300.
3. Adjust meter reading to zero by rotating the Calibrate ADJUST (zero) knob.
4. Attach one end of T assembly to methane gas cylinder and the other to the probe.
5. Crack open methane gas cylinder until a slight flow of gas can be detected exiting the open end of the T assembly.
6. Adjust trimpot R-32 on circuit board so that meter reads the equivalent of the calibration gas concentration. This sets the instrument gain for methane with the panel mounted gain adjustment set at 300.

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7. Close methane gas cylinder. Turn off H<sub>2</sub> SUPPLY VALVE to put out flame. Wait for flameout alarm to sound to ensure the flame is out.
8. Leave CALIBRATE switch on the X10 position and use CALIBRATE ADJUST (zero) knob to adjust meter reading to 4 ppm.
9. Place CALIBRATE switch in X1 position and, using trimpot R-31 on circuit board, adjust meter reading to 4 ppm. This is the Bias Adjustment for the X1 range.
10. Move CALIBRATE switch to X10 position again. Use CALIBRATE ADJUST (zero) knob to adjust meter to a reading of 40 ppm.
11. Move CALIBRATE switch to X100 position and use trimpot R-33 on circuit board to adjust meter reading to 40 ppm.
12. Move CALIBRATE switch to X10 position and use CALIBRATE ADJUST (zero) knob to adjust meter reading to zero.
13. Unit is now balanced from range to range, calibrated to methane, and ready to be placed in normal service.
14. Shut instrument down by ensuring that the H<sub>2</sub> SUPPLY VALVE and H<sub>2</sub> TANK VALVE are closed and the INSTR and PUMP switches are in the OFF position.
15. Record on instrument calibration label, calibration date, gas, and initials of person performing calibration. Remove old tag and replace it with updated label. Fill out instrument history log form.

### 2.3 Calibration to Specialty Gas/Vapor

Primary calibration of the instrument is accomplished using a known mixture of a specific gas or vapor.

#### 2.3.1 Equipment

- o Calibration (span) gas (75-90ppm of known gas or vapor)
- o T-tube assembly

### 2.3.2 Instrument Startup

Follow steps in 2.2.2 above.

### 2.3.3 Calibration

Calibration should be performed in a well ventilated area.

1. Set CALIBRATE switch to 10.
2. Adjust meter reading to zero by rotating the CALIBRATE ADJUST (zero) knob.
3. Attach one end of T assembly to calibration gas cylinder and the other to the probe.
4. Crack open calibration gas cylinder until a slight flow of gas can be detected exiting the open end of the T assembly. (Caution: if the calibration gas is toxic or highly flammable, calibration should occur underneath a hood.)
5. Adjust GAS SELECT knob on instrument until the meter reads the same level as that of the calibration gas.
6. Turn off calibration cylinder and remove T assembly.
7. The instrument is now calibrated for the specialty gas/vapor. All responses of the instrument should be recorded relative to the specialty gas.
8. Calibration in the X10 range by adjusting the GAS Select knob automatically calibrates the instrument for the X1 and X100 ranges. No further adjustments are necessary.
9. Shut instrument down by closing the H<sub>2</sub> SUPPLY VALVE and H<sub>2</sub> TANK VALVE, and putting the INSTR and PUMP switches in the OFF position.
10. Record in instrument calibration label calibration date, span gas and concentration, span setting, and initials of person performing calibration. Remove old

tag and replace it with updated label. Fill out instrument history log.

### 3.0 FILLING OF HYDROGEN SUPPLY

The instrument should be completely shut down for hydrogen tank refilling. The refilling should be done in a ventilated area. There should be no potential ignitors or flame in the area.

1. Attach filling hose to external source of hydrogen. Pure hydrogen of 1,600 to 2,300 psi will be required.
2. Crack open valve on hydrogen supply, place FILL/BLEED valve on hose in FILL position and purge hose for 2-3 seconds.
3. Close FILL/BLEED Valve (OFF position) and immediately attach other end of fill hose to instrument fill connection and tighten the connection.
4. Open supply valve external on hydrogen tank 1/2 to 1 turn. Set regulator to 1,600 to 2,300 psi.
5. Open the REFILL Valve and the H<sub>2</sub> Tank VALVE on the instrument.
6. Place FILL/BLEED Valve in FILL position. The instrument hydrogen tank should now be filling.
7. The instrument H<sub>2</sub> Pressure Indicator should now indicate instrument tank pressure. This pressure should approximate that of the external supply tank regulator gauge.
8. After the instrument tank is filled, shut off:
  - a. The REFILL VALVE on the instrument panel.
  - b. The FILL/BLEED Valve on the filling hose assembly.
  - c. The valve on the external hydrogen supply bottle.
9. The supply hose and internal lines on the instrument now contain hydrogen under pressure. To reduce this pressure to atmospheric pressure:
  - a. Turn FILL/BLEED Valve to BLEED position until gas can no longer be heard escaping.

- b. Turn FILL/BLEED Valve to FILL position to allow gas trapped in the connective fittings to go into the hose assembly.
  - c. Turn FILL/BLEED Valve to BLEED position to bleed off this pressure.
  - d. Turn FILL/BLEED Valve to OFF position. Keep valve in OFF position.
10. Close H<sub>2</sub> Tank Valve.
  11. Remove fill hose from instrument. Any gas still under pressure will escape as the connector is removed. Release of gas should only be momentary.
  12. As a check of the integrity of the instrument's hydrogen supply system, observe the H<sub>2</sub> TANK PRESSURE Indicator with the system shut down. Release of internal pressure should not go down rapidly. If there is a rapid decrease (greater than 350 PSIG/hour) with the instrument in shut down mode, there is a significant leak in the H<sub>2</sub> supply system. If so, the instrument should be returned to the manufacturer for repairs.

#### 4.0 BATTERY RECHARGING

The battery should be recharged following each use of 4 hours or more, or when the battery check indicator indicates need of a charge. Never charge instrument in hazardous environment or when refilling hydrogen tank.

1. Remove cover from battery charge part on instrument.
2. Plug charger BNC connector into mating connector on battery cover and insert AC plug into 115 VAC wall outlet.
3. Move battery charger switch to the ON position. The light above the switch should illuminate.
4. Battery charge condition is indicated by the meter on the front panel of the charger; meter will deflect to the right while charging. When fully charged, the pointer will be in line with the charged mark above the scale.
5. Approximately 1 hour of charging time is required for each hour of operation; 12 hours for complete charge. Do not charge for more than 24 hours.



## 5.0 MAINTENANCE

### 5.1 General

Section 6.0 of the Model OVA-128 Instruction and Service Manual contains detailed maintenance and repair procedures for servicing the OVA. These procedures are not repeated here. Equipment managers are referred to the manual for repair of the OVA.

Equipment managers should be thoroughly familiar with instrument operation before performing maintenance. It is essential that all safety considerations regarding use and maintenance of this instrument be understood. There should be no potential igniters or flame in the area when filling, emptying, or purging the hydrogen system and the instrument should be in shut-down mode.

### 5.2 Trouble Shooting

Table 2 presents common problems and corrective actions for repairing the instrument.

## 6.0 REFERENCE

Foxboro Analytical. No date. Instruction and Service Manual, Century Systems Portable Organic Vapor Analyzer Model OVA-128 (M1 2R900AC).

TABLE 2

TROUBLE	TROUBLE SHOOTING PROCEDURE	REMEDY
1) Low sample flow rate on flow indicator	a) Check teflon tubing on valve assembly for kinks, etc. b) Check flow rate with valve in down position.	Straighten or replace teflon tubing  Check for over restriction of charcoal filter
2) H <sub>2</sub> flame will not light	a) Check column connections on top of unit to make sure they are tight. b) Check column for sharp bends or kinks. (Hydrogen flows through this column at all times and a sharp bend will compact packing too tightly for proper hydrogen flow.) c) Check charcoal filter fittings to make sure they are tight. d) Check H <sub>2</sub> flow rate from the column. e) Check that the Inject and Backflush Valves are both completely in or out. A partially activated valve will block the H <sub>2</sub> and air flow paths. f) If a new column was installed prior to problem identification, check for proper hydrogen flow rate through the column (should be approximately 12 cc/minute). Reference paragraph 7.1.4.2 d.	Tighten fittings  Replace column   Tighten fittings  Adjust hydrogen pressure to obtain 12 cc/min. flow rate. Ensure both valves are either completely in or out.  Increase hydrogen pressure to obtain proper hydrogen flow rate or if column is excessively restrictive, replace or repack the column.
3) Ambient background reading in clean environment is too high	a) Check for contamination in charcoal filter assembly. This can be detected if ambient reading increases when going in to the chromatographic mode. Reference paragraph 7.1.5.2 b. b) Check for contamination in column. Reference paragraph 7.1.5.2 a c) Check for contamination in column valve assembly.	Replace activated charcoal in charcoal filter assembly.   Replace or clean column.  Remove valve stems and wipe with clean lint-free cloth. Heat valve assembly during operation to vaporize and remove contaminants.
4) Flame-out when operating either valve	a) Ensure valves are being operated with a quick, positive motion. b) Either H <sub>2</sub> or air may be leaking around one or more of the valve quad rings. Assess by tests and "O" ring inspection. c) Damaged or worn quad rings causing leak.	Operate valve with a positive motion.  Remove stems and lightly coat with silicone grease, only on contact surface of the "O" ring. Wipe off excess (do not remove quad rings). Replace quad rings and grease as above.
5) Excessive peak tailing	a) Change or clean GC column and see if problem disappears.  b) Inspect GC valves for excessive silicone grease or contamination.	Ensure columns are clean prior to use. Refer to paragraph 7.1.5.2 a for cleaning instructions. If one of a same type of column tails worse than others, repack the column or discard. Excessive lubricant or foreign matter in the valve assembly can cause excessive tailing. Clean valve assemblies and lightly relubricate as required. Lubricant should be put only on the outside contact surface of the "O" ring. Do not get grease into "O" ring

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CALIBRATION AND MAINTENANCE PROCEDURE  
YSI MODEL 33 S-C-T METER

1.0 INTRODUCTION

This procedure presents steps to calibrate and maintain the YSI Model 33 S-C-T meter. Operation principles, procedures, and equipment specifications are presented in Procedure 5617002 and are not repeated here.

2.0 CALIBRATION

2.1 Temperature

2.1.1 Temperature Knob Setting

It is possible for the temperature knob to become loose or slip from its normal position. In an emergency, the dial can be repositioned. It must be emphasized that this is an emergency procedure only and that the instrument should be returned to the factory for proper recalibration - at the earliest opportunity.

To recalibrate the temperature setting:

1. Red line instrument and then place probe in sample of known conductivity.
2. Read and record the temperature and conductivity of the solution using appropriate settings. Leave probe in solution.
3. Determine the salinity of the solution by running a line vertically on Figure 1 until it intersects the appropriate 'C' line. From this intersection, extend a line horizontally to the left edge of the graph (Figure 1). This determines the salinity of the sample.

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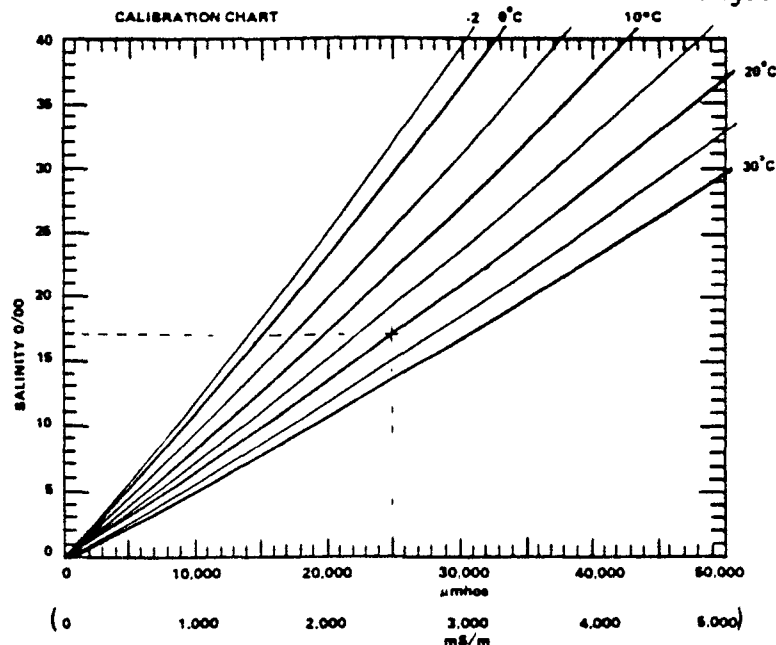


Figure 1. Calibration Chart for Resetting Temperature Knob

4. Remove the 'C knob switch to SALINITY, and turn the control shaft until the meter needle indicates the salinity value determined in step 3.
5. Switch to TEMPERATURE. If this temperature is the same as step 2, continue. If not, repeat steps 1 through 5.
6. Place the knob on the control shaft - without turning the control shaft - with the pointer at the same temperature as the meter reading. Tighten both sets of screws securely. Care must be taken at this step so that the shaft setting is not moved.
7. Return the instrument to the factory at the earliest opportunity.

#### 2.1.2 Temperature Probe/Instrument

To check the accuracy of the Probe/Instrument temperature readings:

1. Place NBS traceable thermometer in solution with thermometer and probe.
2. Place instrument in temperature mode after zeroing and red lining the instrument.

3. After five minutes, compare temperature of thermometer and instrument. If the instrument varies by  $\pm 1^{\circ}\text{C}$ , the instrument should be returned to the factory for calibration and maintenance.

## 2.2 Probe Cell Calibration

The YSI #3300 Series Cells are calibrated to absolute accuracy of  $\pm 1.5$  percent based on a standard solution of 0.01  $\text{mol/L}$  KCl. To prepare this solution:

1. In a one liter flask, dissolve 0.745 grams of pure dry KCl until the solution is one kilogram in weight.
2. Use Table 1 and the temperature of the water to determine the conductivity of the solution just prepared. Note: Table 1 shows conductivity as if the distilled water was nonconductive. Since even high purity distilled water is slightly conductive, the measured conductivity will be higher by an amount equal to the water's conductivity.
3. Place probe in solution and measure conductivity. The conductivity of the solution plus the conductivity of the distilled water should not vary from the meter reading by  $\pm 1.5\%$ . If the reading is greater than  $1.5\%$ , clean the probe and then recheck the conductivity. If after cleaning it is not possible to measure the conductivity of the calibration solution within  $\pm 1.5\%$ , the probe and instrument should be returned to the manufacturer for calibration and maintenance.

## 3.0 MAINTENANCE

### 3.1 Batteries

The batteries should be replaced either (1) when it is not possible to red line the instrument, (2) after 200 hours of operation, or (3) every 6 months to reduce the danger of corrosion due to leaky batteries.

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To replace batteries, remove the six screws from the rear plate. The battery holders are color coded. The positive (+ button) end must go on red.

Use two "D" size alkaline flashlight cells (Eveready E95 or equivalent).

### 3.2 Probe

#### 3.2.1 Cleaning

When the cell test indicates low readings, the probable cause is dirty electrodes. Hard water deposits, oils, and organic matter are the most likely contaminants.

TABLE 1 - CELL CALIBRATION DATA

Temperature (°C)	Conductivity (umhos/cm)
15	1141.5
16	1167.5
17	1193.6
18	1219.9
19	1246.4
20	1273.0
21	1299.7
22	1326.6
23	1353.6
24	1380.8
25	1408.1
26	1436.5
27	1463.2
28	1490.9
29	1518.7
30	1546.7

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For convenient normal cleaning, soak the electrodes for 5 minutes with a locally available bathroom tile cleaner such as: "Rally, Tile, Porcelain, and Chrome Cleaner"; Johnson Wax "Envy, Instant Cleaner"; or Lysol Brand "Basin, Tub, Tile Cleaner".

For storage cleaning, a 5 minute soak in a solution made of 10 parts distilled water, 10 parts isopropyl alcohol, and 1 part HCl can be used.

Always rinse the probe in distilled water after cleaning and before storage.

CAUTION: Do not touch the electrodes inside the probe. Platinum black is very soft and can be scraped off.

If cleaning does not restore the probe performance, re-platinizing is required.

### 3.2.2 Probe Replatinizing

#### 1. Equipment required:

- a. YSI #3140 Platinizing Solution, 2 fluid ounce (3% platinum chloride dissolved in 0.025% lead acetate solution)
- b. YSI Model 33 meter
- c. 50 ml glass beaker or equivalent
- d. Distilled water

#### 2. Procedure

- a. Clean probe as in section 3.2.1 - either method

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- b. Place the cell in the beaker and add sufficient YSI #3140 solution to cover the electrodes. Do not cover the top of the probe
- c. Plug the probe into the Model 33 and switch to the X100 scale to platinize the electrode

- d. Move the probe slightly to obtain the highest meter reading and continue platinizing for the appropriate time shown below:

<u>Meter Reading</u> (umhos/cm)	<u>Time</u> (minutes)
30,000	5
25,000	6
20,000	8
15,000	11
10,000	16

- e. After the elapsed time, remove the probe and rinse in distilled water.
- f. Return the solution to its container. Two ounces of solution should be sufficient for 50 treatments.

### 3.2.3 Storage

It is best to store conductivity cells in deionized water. Cells stored in water require less frequent platinization. Any cell that has been stored dry should be soaked in deionized water for 24 hours before use.



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## CALIBRATION AND MAINTENANCE PROCEDURES HAAKEBUCHLER pH STICK

### 1.0 INTRODUCTION

This procedure presents the steps for calibrating and maintaining the HaakeBuchler pH Stick. Instrument operation principles and procedures and specifications are presented in Procedure 5617003.

### 2.0 CALIBRATION

#### 2.1 Calibration Solutions

The instrument requires distilled water, a pH 7 buffer solution, and a pH 4 buffer solution for calibration. To prepare the buffer solutions, dissolve the buffer powders provided with the instrument into the volume of distilled water specified on the buffer powder packets. (Note: the manufacturer does not specify whether buffer and pH 4 solutions, other than that provided, may be used as substitute solutions).

The pH of the buffer and pH 4 solutions will vary with the temperature of the solution. Use the table below to determine solution pH based on temperature.

Temp	0°C	10°C	20°C	25°C	30°C	40°C	50°C
pH 4	4.00	4.00	4.00	4.01	4.02	4.04	4.06
pH 7	7.11	7.06	7.01	7.00	6.98	6.97	6.97

#### 2.2 Calibration Procedure

The instrument requires calibration in the field prior to each use. However, as a check of proper instrument function, the instrument should be periodically calibrated in the laboratory,

particularly if the instrument has been stored for an extended period without use.

To calibrate the instrument:

1. Remove the protective sheath and rinse the electrode in distilled water.
2. Place the electrode in the pH 7 buffer solution, depress the white operation button below the LCD display and allow the reading to stabilize.
3. Adjust pH 7 control using the tool on the end of the protective sheath. The pH 7 control is the upper most white control on the right side of the instrument. Adjust the pH control until the meter reads pH 7.
4. Rinse the electrode in distilled water.
5. Place the electrode in pH 4 solution, depress the white operation button, and allow the reading to stabilize.
6. Adjust the slope control (white control below pH 7 control on the right side of the instrument) until the meter reads the correct value of the pH 4 solution.
7. Rinse the probe in distilled water.
8. Repeat steps 2 through 7.
9. Record calibration on the instrument log form.
10. Store instrument properly.

### 3.0 MAINTENANCE

#### 3.1 Storage

To maintain high accuracy and to obtain a long electrode life, the pH stick must be stored correctly when not in use. Always rinse the electrode in distilled water before replacing it in its protective sheath. The electrode must not be let to dry out.

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The absorbent pad at the bottom of the sheath must be kept saturated with a pH 7 buffer solution. If this is not available, distilled water can be used as a temporary measure. Replace distilled water with buffer solution at the earliest possible opportunity. Always place buffer (or distilled water) into sheath following each use.

To retain accuracy and speed of response, the insulation of the connectors on the electrode and the body must be kept clean and dry. This is best assured by not unnecessarily removing the electrode from the body.

When not in use, place the pH stick in the wallet provided and store in a dry place.

### 3.2 Electrode Cleaning

If rinsing the electrode in distilled water is not deemed sufficient to clean the electrode, it can be cleaned in a N/10 HCl acid solution. Following cleaning in the acid, the electrode should be soaked in a pH 7 buffer solution for 24 hours before rinsing. Record cleaning on instrument's log form.

### 3.3 Battery

Normal battery life is in excess of 200 hours of continuous use. Cells should be replaced at 2 year intervals or earlier if exhausted (voltage per cell of less than 1.35V). Replacement cells must be mercury type V312H or direct equivalent. When refitting cells, make sure they are refitted in the manner illustrated on the battery housing.